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29 December 1953
Report No. 770
(Quarterly)
Copy No. A

EXPLOSIVES RESEARCH



Contract N7onr-46208

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29 December 1953

Report No. 770
(Quarterly)

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EXPLOSIVES RESEARCH

Contract N7onr-46208

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AEROJET-GENERAL CORPORATION

Anaheim, California

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CONTRACT FULFILLMENT STATEMENT

This quarterly report is submitted in partial fulfillment of
Contract N7onr-46208.

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I. SUMMARY

A. This quarterly report is submitted under Contract N7onr-46208 and covers the period 1 August through 31 October 1953.* The objectives** of the contract are threefold:

1. Synthesis and physical studies of new high-energy polynitro compounds as potential explosives.
2. Desensitization of RDX with materials that will not lower the oxygen balance appreciably.
3. Development of bomb and shell casings made of an explosive plastic.

B. The more important results and conclusions are presented below:

1. Three polynitrocaramates were synthesized:
 - a. 2,2,9,9,16,16-Hexanitro-5,13-diaza-6,12-diketo-7,11-dioxa-heptadecane (I) from 3,3-dinitrobutyl isocyanate and 2,2-dinitro-1,3-propanediol.
 - b. 1,1,1-Trinitro-3-oxa-4-keto-5-aza-heptane (II) from ethyl isocyanate and 2,2,2-trinitroethanol.
 - c. 1,1,1,8,11,18,18,18-Octanitro-3,16-dioxa-4,15-diketo-5,8,11,14-tetraza-octadecane (III), from 3,6-dinitraza-1,8-octane diisocyanate and 2,2,2-trinitroethanol.
2. Three postnitrated polynitrocaramates were prepared:
 - a. 2,2,5,9,9,13,16,16-Octanitro-5,13-diaza-6,12-diketo-7,11-dioxa-heptadecane (IV) by the nitration of I.
 - b. 1,1,1,5-Tetranitro-3-oxa-4-keto-5-aza-heptane (V) by the nitration of II.

*

Previous work on this contract was covered in Aerojet Reports No. 512, 538, 562, 589, 621, 637, 660, 682, 711, and 737.

**

Recently a conference was held with representatives of the Bureau of Ordnance and the objectives of the contract were changed. In the future, emphasis will be placed on developing new synthetic methods for the preparation of explosives and new types of explosive compounds. Work on the desensitization of RDX will be discontinued, and the development of bomb and shell casings made of an explosive plastic will be postponed.

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I Summary, B (cont.)

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c. 1,1,1,5,8,11,14,18,18,18-Decanitro-3,16-dioxa-4,15-diketo-5,8,11,14-tetraza-octadecane (VI) by the nitration of III.

3. Two aliphatic secondary polynitronitramines were prepared.

a. Condensation of 2,2,2-trinitroethanol and 3,6-dinitraza-1,8-octane diamine gave 1,1,1,6,9,14,14,14-octanitro-3,6,9,12-tetraza-tetradecane (VII). Nitration of VII yielded 1,1,1,3,6,9,12,14,14-decanitro-3,6,9,12-tetraza-tetradecane (VIII).

b. Condensation of 2,2,2-trinitroethanol and 3-nitraza-1,5-pentane diamine gave 1,1,1,6,11,11,11-heptanitro-3,6,9-triaza-undecane (IX). Nitration of IX yielded 1,1,1,3,6,9,11,11,11-nonanitro-3,6,9-triaza-undecane (X).

4. N,N'-bis(3,3-Dinitrobutyl) oxamide (XXI) was prepared by the condensation of 3,3-dinitrobutyl amine and ethyl oxalate. Nitration of XXI gave N,N'-dinitro-N,N'-bis(3,3-dinitrobutyl) oxamide (XXII).

5. Four 2,2,2-trinitroethyl esters were prepared from the corresponding acid chloride, 2,2,2-trinitroethanol, and a catalytic amount of anhydrous aluminum chloride.

a. bis(2,2,2-Trinitroethyl)-4,4-dinitroheptane-dioate (XXIII).

b. bis(2,2,2-Trinitroethyl)-4-nitraza-heptanedioate (XXIV).

c. bis(2,2,2-Trinitroethyl)-4,7-dinitraza-decane-dioate (XXV).

d. 2,2,2-Trinitroethyl-3,5,5-trinitro-3-aza-hexanoate (XXVI).

6. An alternate synthesis of 1-nitrato-3,5,5-trinitro-3-aza-hexane (XXVIII) was made and its structure confirmed.

7. 2,4,4-Trinitro-2-aza-amyl isocyanate (XXX) was isolated, purified, and proved to be extremely unstable and dangerous at room temperature.

8. Twelve of the new compounds were submitted to the Naval Ordnance Laboratory for evaluation as potential explosives. The preliminary evaluation of many of these compounds is described.

9. SPIA data sheets have been completed on 16 compounds and are included in the appendix.

10. A summary of the heats of formation and oxygen contents of the explosive compounds prepared on this program has been compiled.

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II. TECHNICAL PROGRESS: SYNTHESIS OF NEW HIGH EXPLOSIVES

A. INTRODUCTION

1. The present explosives program is directed toward the synthesis of new, stable, high-energy polynitro compounds with a preferred oxygen balance on the positive side or very near zero on the negative side.

2. This report describes the synthesis of aliphatic polynitrocaramates, polynitronitramines, polynitronitramides, polynitroesters, and polynitronitrates.

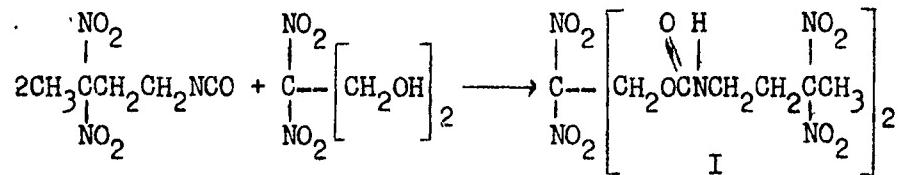
B. PREPARATION OF ALIPHATIC POLYNITROCARBAMATES

1. Discussion

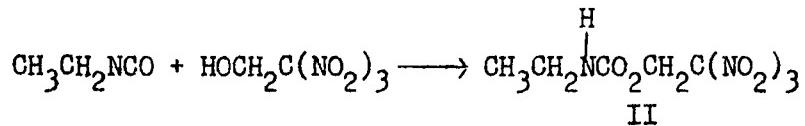
The preparation of aliphatic polynitrocarbamates for evaluation as high explosives has been previously reported.* This work is being continued.

a. The following aliphatic polynitrocarbamates were synthesized:

(1) 2,2,9,9,16,16-Hexanitro-5,13-diaza-6,12-diketo-7,11-dioxa-heptadecane (I) from 3,3-dinitrobutyl isocyanate and 2,2-dinitro-1,3-propanediol:



(2) 1,1,1-Trinitro-3-oxa-4-keto-5-aza-heptane (II) from ethyl isocyanate and 2,2,2-trinitroethanol:



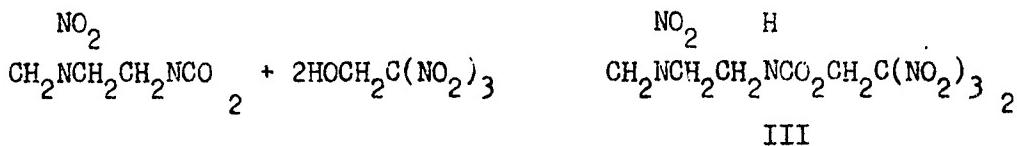
(3) 1,1,1,8,11,18,18,18-Octanitro-3,16-dioxa-4,15-diketo-5,8,11,14-tetraza-octadecane (III) was prepared by the addition of two moles of 2,2,2-trinitroethanol to one mole of 3,6-dimitraza-1,8-octane diisocyanate:

* Aerojet Reports No. 562, 589, 621, and 737.

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II Technical Progress, B (cont.)

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b. Two new alcohols, 2-nitroto-ethanol and 2-nitramino-ethanol, were investigated as intermediates for the synthesis of high-energy polynitrocaramates. These alcohols were treated with 3,3,3-trinitropropyl isocyanate and 3,3,5,7,7-pentanitro-5-aza-1,9-nonane diisocyanate to give oils. Crystalline solids could not be obtained by the nitration of these oils. This is in contrast to the many solid post-nitrated polynitrocaramates that have been prepared from such intermediate oils.

2. Experimental

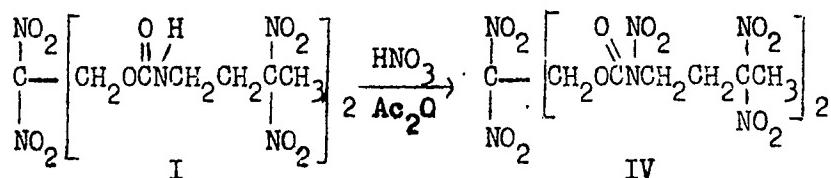
The general procedure employed was to reflux equivalent quantities of the alcohol and isocyanate with a catalytic amount of ferric acetylacetone for 6 to 8 hours in dry chloroform or ethylene dichloride solution, and concentrate in vacuo. The results are summarized in Table I.

C. PREPARATION OF POSTNITRATED ALIPHATIC POLYNITROCARAMATES

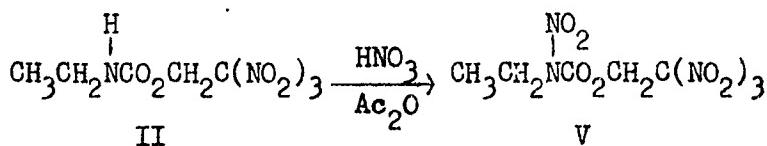
1. Discussion

The following postnitrated aliphatic polynitrocaramates were prepared by the nitration of the nitrocaramates described in II,B with a mixture of 100% nitric acid and acetic anhydride at 5 to 10°C.

a. 2,2,5,9,9,13,16,16-Octanitro-5,13-diaza-6,12-diketo-7,11-dioxa-heptadecane (IV):



b. 1,1,1,5-Tetranitro-3-oxa-4-keto-5-aza-heptane (V):



c. 1,1,1,5,8,11,14,18,18,18-Decanitro-3,16-dioxa-4,15-diketo-5,8,11,14-tetraza-octadecane (VI):

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II Technical Progress, C (cont.)

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TABLE I
ALIPHATIC POLYNITROCARBAMATES

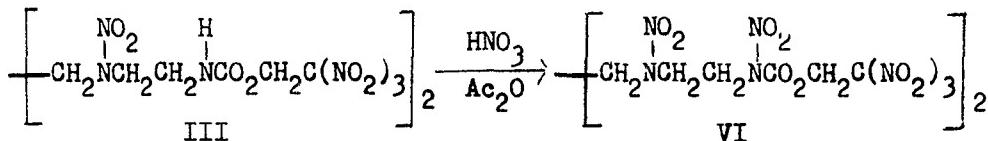
Isocyanate	$\text{CH}_3\text{CH}_2\text{NCO}$	$\left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2\text{NCH}_2\text{CH}_2\text{NCO}_2 \end{array} \right]_2$	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	$\left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2\text{NCH}_2\text{CH}_2\text{NCO}_2\text{CH}_2\text{NCO}_2 \end{array} \right]_2$
Alcohol	$\text{NO}_2 \left[\begin{array}{c} \text{CH}_2\text{OH} \\ \\ \text{C}-\text{NO}_2 \end{array} \right]_2$	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	$\text{H} \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_3\text{CH}_2\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3 \end{array} \right]_2$	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$
Polynitrocarbamate	$\text{NO}_2 \left[\begin{array}{c} \text{OH} \\ \\ \text{CH}_2\text{O}\text{CNCH}_2\text{CH}_2\text{C}(\text{NO}_2)_3 \end{array} \right]_2$	$\text{CH}_3\text{CH}_2\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$	Quant.	Quant.
Yield, %	Quant.	128-129	85.7	150-158
mp, °C		oil		
Recrystallization Solvent	Ethylenic Dichloride	-----	-----	-----
Formula	$\text{C}_{13}\text{H}_{20}\text{N}_4\text{O}_8$	$\text{C}_5\text{H}_8\text{N}_4\text{O}_8$	$\text{C}_{12}\text{H}_{18}\text{N}_4\text{O}_8$	
Analyses, %	Calcd. C 28.68 H 3.70 N 20.59	Found C 29.13 H 3.91 N 20.82		

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2. Experimental

a. The general procedure used was to cool the 100% nitric acid to 0 to 5°C and add the acetic anhydride dropwise, keeping the temperature below 10°C. The solid nitrocarbamate was added portionwise at 5 to 10°C. (Ten ml of 100% nitric acid and 10 ml of acetic anhydride was used per gram of nitrocarbamate.) The solid dissolved readily, and the solution was stirred for 20 min at 5 to 10°C and poured on ice. The white solid was collected, washed with water, and dried in vacuo over potassium hydroxide.

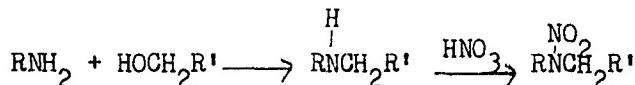
b. In the instance in which the nitrocarbamate was an oil (compound II), the oil was dissolved in the acetic anhydride and the solution was added dropwise to the 100% nitric acid, keeping the temperature at 5 to 10°C.

c. The experimental results are summarized in Table II.

D. PREPARATION OF ALIPHATIC SECONDARY POLYNITRONITRAMINES

1. Introduction

The preparation of aliphatic secondary polynitronitramines for evaluation as high explosives has been continued.* This class of compounds, which is high in explosive power, is prepared by the Mannich condensation of a polynitro-alcohol and a polynitroamine, followed by nitration of the secondary amine:

2. Preparation of 1,1,1,3,6,9,12,14,14,14-Decanitro-3,6,9,12-tetraza-tetradecane

a. Discussion

(1) The condensation of two moles of 2,2,2-trinitro-ethanol with one mole of 3,6-dinitraza-1,8-octane diamine gave 1,1,1,6,9,14,14,14-octanitro-3,6,9,12-tetraza-tetradecane (VII), a bright yellow solid, mp 100 to 105°C dec. Nitration of VII with a mixture of acetic anhydride and 100% nitric acid at low temperatures gave a gummy solid which was difficult to purify. However, when VII was nitrated with a mixture of concentrated sulfuric acid and 100% nitric acid at 50°C, 1,1,1,3,6,9,12,14,14,14-decanitro-3,6,9,12-tetraza-tetradecane (VIII) was formed. VIII is a white crystalline solid, mp 166 to 167°C, with a calculated lead-block value of 162 and a calculated ballistic-mortar value of 156:

* For previous work see Aerojet Reports No. 621, 660, and 682.

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TABLE II
POSTNITRATED ALIPHATIC POLYNITROCARBAMATES

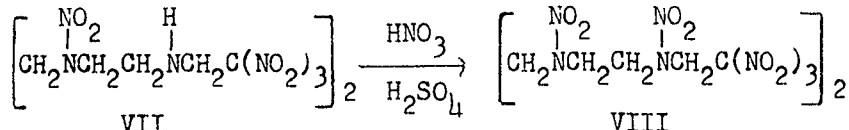
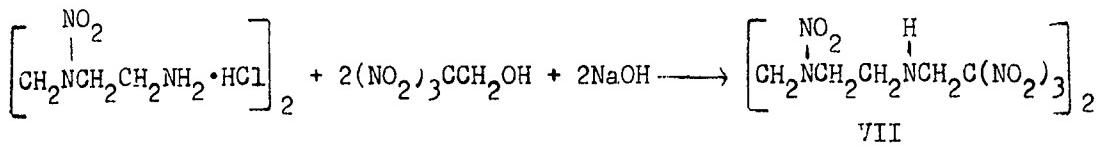
Postnitrated Polynitrocaramate	Yield, %	mp, °C	Recryst. Solvent	Formula	Analyses, %	Calc'd.	Found	Calc'd. Lead-Block Value (TNT = 100)	Calc'd. Ballistic-Mortar Value (TNT = 100)
$\text{NO}_2 \text{---} \text{C}(\text{NO}_2)_2 \text{---} \text{CH}_2 \text{OCNCH}_2 \text{---} \text{CH}_2 \text{C}(\text{NO}_2)_3 \text{---} \text{NO}_2$	88	117-118	Ethylene Dichloride	$\text{C}_{13}\text{H}_{18}\text{N}_{10}\text{O}_{20}$		24.61 2.86 22.08	24.78 2.90 21.87	119	128
$\text{NO}_2 \text{---} \text{C}(\text{NO}_2)_2 \text{---} \text{CH}_2 \text{OCNCH}_2 \text{---} \text{CH}_2 \text{C}(\text{NO}_2)_3 \text{---} \text{NO}_2$	85.7	50-51	Carbon Tetrachloride	$\text{C}_5\text{H}_7\text{N}_5\text{O}_{10}$		20.21 2.38 23.57	20.55 2.04 23.68	146	136
$\text{NO}_2 \text{---} \text{C}(\text{NO}_2)_2 \text{---} \text{CH}_2 \text{OCNCH}_2 \text{---} \text{CH}_2 \text{C}(\text{NO}_2)_3 \text{---} \text{NO}_2$	82.5	127-128	70% Nitric Acid	$\text{C}_{12}\text{H}_{16}\text{N}_4\text{O}_{24}$		19.47 2.18 26.49	19.51 2.29 25.78	142	141
$\text{NO}_2 \text{---} \text{C}(\text{NO}_2)_2 \text{---} \text{CH}_2 \text{OCNCH}_2 \text{---} \text{CH}_2 \text{C}(\text{NO}_2)_3 \text{---} \text{NO}_2$									

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(2) Compound VIII was purified by recrystallization from concentrated nitric acid, and the analyses were in close agreement with the calculated values. However, it has been noted in the past that solids recrystallized from nitric acid usually retain a small amount of occluded solvent, which catalyzes the decomposition of the compound during thermal stability tests at elevated temperatures. Thus, a true criterion of the actual thermal stability may not be obtained. For this reason, a solvent other than nitric acid was sought for the purification of VIII. It was found that the product, after recrystallization from nitric acid, could be recrystallized from acetone, raising the melting point from 166 to 167°C dec to 167 to 169°C dec. However, the analysis and heat of combustion values for the acetone-purified product differed greatly from the calculated values. No ready explanation was apparent for this behavior.

b. Experimental

(1) A solution of 69.5 g (0.38 mole) of 2,2,2-trinitroethanol in 250 ml of water and a solution of 17.0 g (0.19 mole) of 3,6-dinitraza-1,8-octane diamine dihydrochloride in 250 ml of water was placed in a one-liter three-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel. Then a solution of 15.3 g (0.38 mole) of sodium hydroxide in 100 ml of water was added dropwise in 25 min, the temperature rising from 23 to 25°C. The bright-yellow solid which precipitated was collected and dried in vacuo over potassium hydroxide. The yield of crude product was quantitative, mp 100 to 105°C dec.

(2) A mixture of 500 ml concentrated sulfuric acid and 550 ml of 100% nitric acid was cooled to 20°C and, with good stirring, 56.2 g (0.1 mole) of the above-mentioned yellow solid was added. The reaction mixture was heated to 50°C within 30 min, held at this temperature for 20 min, cooled, and poured on ice. The white solid was collected, washed with water, and dried in vacuo over potassium hydroxide. The yield of crude product was quantitative, mp 149 to 155°C. Three recrystallizations from concentrated nitric acid gave white needles, melting at 166 to 167°C.

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Anal. Calc'd for $C_{10}H_{16}N_{14}O_{20}$: %C, 18.41; %H, 2.47; %N, 30.06

Found: %C, 18.22; %H, 2.38; %N, 29.86

Heat of Combustion, ΔH_p , Predicted: 2321 cal/g

Found: 2222 cal/g

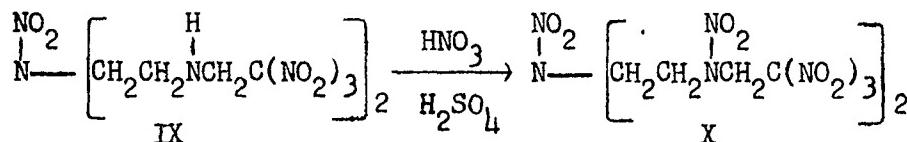
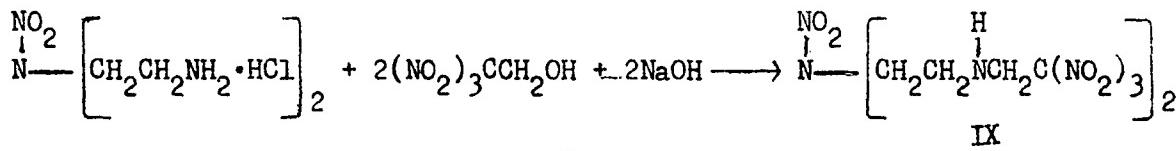
Recrystallization of the above compound from acetone gave a white solid, mp 167 to 169°C.

Anal. Found: %C, 20.76; %H, 2.79; %N, 28.37; ΔH_p , 2583 cal/g.

3. Preparation of 1,1,1,3,6,9,11,11,11-Nonanitro-3,6,9-triaza-undecane

a. Discussion

The condensation of one mole of 3-nitraza-1,5-pentane diamine and two moles of 2,2,2-trinitroethanol yielded 1,1,1,6,11,11,11-heptanitro-3,6,9-triaza-undecane (IX), a yellow gummy solid. Nitration of IX with a mixture of 100% nitric acid and concentrated sulfuric acid at 50°C gave 1,1,1,3,6,9,11,11,11-nonanitro-3,6,9-triaza-undecane (X), a white crystalline solid, mp 157 to 158°C, with a calculated lead-block value of 175 and a calculated ballistic-mortar value of 157:



Compound X was recrystallized both from concentrated nitric acid and a mixed solvent of acetone and chloroform. In both cases the analyses checked well with the theoretical values.

b. Experimental

(1) A 22.1-g (0.1 mole) quantity of 3-nitraza-1,5-pentane diamine dihydrochloride, 36.2 g (0.2 mole) of 2,2,2-trinitroethanol, and 75 ml of water was placed in a 500-ml three-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel. A solution of 8.0 g (0.2 mole) of sodium hydroxide in 50 ml of water was added dropwise at room temperature. A yellow, gummy solid was precipitated, collected, and dried; the weight was 15.7 g.

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II Technical Discussion, D (cont.)

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(2) A mixture of 150 ml of concentrated sulfuric acid and 175 ml of 100% nitric acid was cooled to 20°C and, with good stirring, 15.7 g of the above-mentioned yellow, gummy solid was added. The reaction mixture was heated to 50°C, held at this temperature for 30 min, cooled, and soured on ice. The white solid was collected, washed with water, and dried in vacuo over potassium hydroxide to give 5.0 g of product. Two recrystallizations from concentrated nitric acid gave white needles, mp 157 to 158°C dec.

Anal. Calc'd for $C_8H_{12}N_{12}O_{18}$: %C, 17.03; %H, 2.14; %N, 29.79

Found: %C, 17.57; %H, 2.38; %N, 29.78

Recrystallization from a mixture of acetone and chloroform raised the melting point to 158 to 159°C.

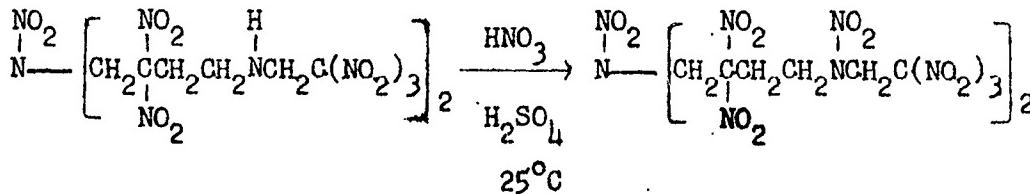
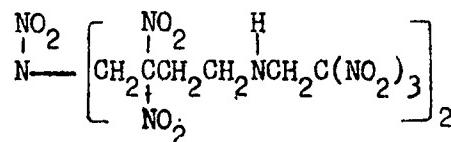
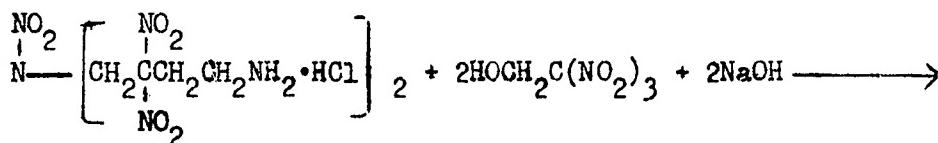
Anal. Calc'd for $C_8H_{12}N_{12}O_{18}$: %C, 17.03, %H, 2.14; %N, 29.79

Found: %C, 17.47; %H, 2.46; %N, 29.88

4. Attempted Preparation of 1,1,1,3,6,6,8,10,10,13,15,15,15-Tridecanitro-3,8,13-triaza-pentadecane

a. Discussion

The condensation of one mole of 3,3,5,7,7-pantanitro-5-aza-1,9-nonane diamine and two moles of 2,2,2-trinitroethanol gave a yellow solid, mp 105 to 118°C dec. Nitration of this condensation product with a mixture of 100% nitric acid and concentrated sulfuric acid at 25°C gave a white solid, mp 90 to 110°C dec. All attempts to purify this product were unsuccessful.



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b. Experimental

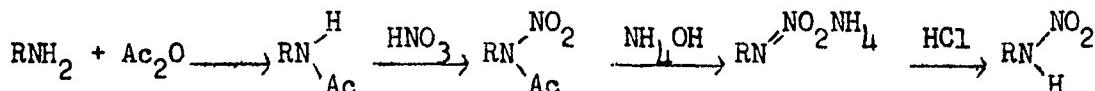
(1) A 22.8-g (0.05 mole) quantity of 3,3,5,7,7-pentanitro-5-aza-1,9-nonane diamine dihydrochloride, 18.1 g (0.1 mole) of 2,2,2-trinitroethanol, and 75 ml of water was placed in a 500-ml, three-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel. A solution of 4.0 g (0.1 mole) of sodium hydroxide in 25 ml of water was added dropwise at room temperature. A yellow solid was precipitated, collected, and dried; the weight was 17.0 g, and the mp was 105 to 118°C dec.

(2) A mixture of 40 ml of 100% nitric acid and 40 ml of concentrated sulfuric acid was cooled to 25°C and, with good stirring, 4.0 g of the above-mentioned yellow solid was added. The reaction mixture was stirred for 20 min at 25°C and poured on ice. The white solid was collected, washed with water, and dried; the weight was 4.3 g, and the mp was 90 to 110°C dec. All attempts to purify this product were unsuccessful.

E. ATTEMPTED PREPARATION OF PRIMARY POLYNITRONITRAMINES

1. Introduction

One of the generally used procedures for the preparation of primary nitramines is as follows:



This method is successful where R is an alkyl group and even when R contains a gem dinitromethyl group (cf. 3,3-dinitro-1,5-pentane-dinitramine, Aerojet Report No. 621, p. 11). The present work was concerned with the attempted conversion of 3,3-dinitro-butyl amine, 3-nitraza-1,5-pentane diamine, and 3,6-dinitraza-1,8-octane diamine to the corresponding nitramines.

2. Attempted Preparation of 3,3-Dinitrobutyl Nitramine

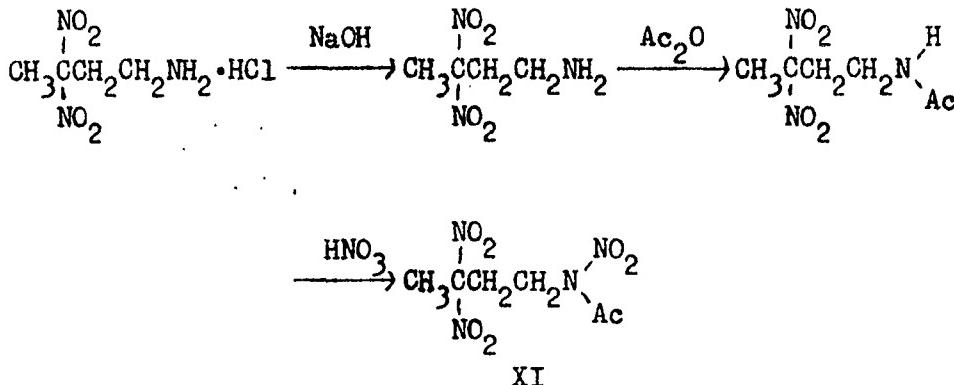
a. Discussion

3,3-Dinitrobutyl amine hydrochloride was converted to the free amine, acetylated, and nitrated to give N-nitro-N-acetyl 3,3-dinitrobutyl amine (XI), a white crystalline solid, mp 79 to 80°C.

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Hydrolysis of XI with ammonium hydroxide, followed by acidification, gave a yellow oil. The analysis of this oil did not correspond to that of any postulated structure.

b. Experimental

(1) Forty grams (0.2 mole) of 3,3-dinitrobutyl amine hydrochloride, 75 ml of water, and 250 ml of ether was placed in a 500-ml, three-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel. The flask was cooled in an ice bath, and a solution of 8.0 g (0.2 mole) of sodium hydroxide in 25 ml of water was added dropwise. The mixture was stirred for 30 min, the ether layer was separated, and the mixture was dried. The ether solution of the free amine was cooled in an ice bath, and 100 ml of acetic anhydride was added. A vigorous reaction occurred, with an initial separation of white solid which dissolved as more acetic anhydride was added. The solution was refluxed for one hour, cooled, and added dropwise to 200 ml of 100% nitric acid, keeping the temperature below 10°C. The solution was poured on ice, precipitating a white solid which was collected and dried, 27.5 g (55%), mp 79 to 80°C. Recrystallization from methanol did not raise the melting point.

Anal. Calc'd for $\text{C}_6\text{H}_{10}\text{N}_4\text{O}_7$: %C, 28.80; %H, 4.03; %N, 22.40

Found: %C, 29.37; %H, 4.28; %N, 22.27

(2) One gram of N-nitro-N-acetyl-3,3-dinitrobutyl amine and 10 ml of concentrated ammonium hydroxide was warmed on a steam bath. The yellow solution was cooled, acidified with dilute hydrochloric acid, and extracted with ether. The ether solution was dried, and concentrated to give 0.8 g of yellow oil, which was distilled twice from a bulb tube, bp 80 to 85°C (4μ), n_D^{26} 1.4667.

Anal. Calc'd for $\text{C}_4\text{H}_8\text{N}_4\text{O}_6$: %C, 23.08; %H, 3.88; %N, 26.92

Found: %C, 27.66; %H, 4.41; %N, 17.50

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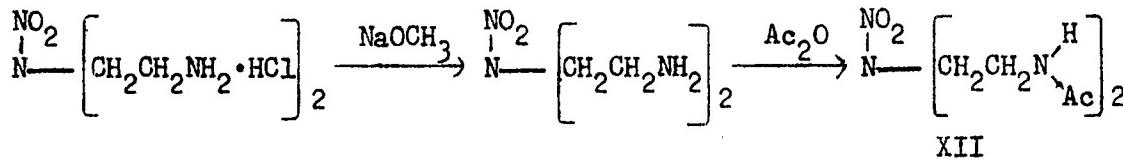
II Technical Progress, E (cont.)

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3. Attempted Preparation of 3-Nitraza-1,5-pentane Dinitramine

a. Discussion

3-Nitraza-1,5-pentane diamine dihydrochloride was converted to the free diamine and acetylated to give N,N'-diacetyl-3-nitraza-1,5-pentane diamine (XII), a white crystalline solid, mp 197 to 199°C.



However, nitration of XII did not give the desired N,N'-diacetyl N,N'-dinitro-3-nitraza-1,5-pentane diamine but instead water-soluble products.

b. Experimental

A 1.4069 N solution of sodium methoxide in methanol (142.2 ml, 0.2 mole) was added dropwise with stirring at 0 to 5°C to a suspension of 22.1 g (0.1 mole) of 3-nitraza-1,5-pentane diamine dihydrochloride in 50 ml absolute methanol. The mixture was stirred for 30 min at 0 to 5°C, and the precipitate of sodium chloride was removed by filtration and washed with methanol. The solution was concentrated in vacuo and diluted with 125 ml of methylene dichloride. To this solution was added portionwise 30 ml of acetic anhydride. A vigorous reaction occurred with a white solid precipitating. The reaction mixture was refluxed for 1 hr, cooled, and filtered. The yield was 14.9 g (64.3%), mp 197 to 198°C. Recrystallization from methanol did not raise the melting point.

Anal. Calc'd for C₈H₁₆N₄O₄: %C, 41.37; %H, 6.95; %N, 24.13

Found: %C, 41.78; %H, 6.97; %N, 23.73

(2) N,N'-Diacetyl-3-nitraza-1,5-pentane diamine was dissolved in acetic anhydride and added to 100% nitric acid, keeping the temperature below 10°C. When the acid solution was poured on ice, only water-soluble products were obtained.

4. Attempted Preparation of 3,6-Dinitraza-1,8-octane Dinitramine

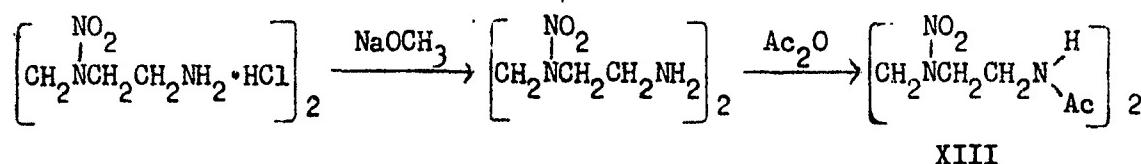
a. Discussion

3,6-Dinitraza-1,8-octane diamine dihydrochloride was converted to the free diamine and acetylated to give N,N'-diacetyl-3,6-dinitraza-1,8-octane diamine (XIII), a white crystalline solid, mp 178 to 180°C.

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II Technical Progress, E (cont.)

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Nitration of XIII also gave water-soluble products.

b. Experimental

(1) A 1.4069 N solution of sodium methoxide in methanol (142.2 ml, 0.2 mole) was added dropwise with stirring at 0 to 5°C to a suspension of 30.9 g (0.1 mole) of 3,6-dinitraza-1,8-octane diamine dihydrochloride in 75 ml of methanol. The mixture was stirred for 30 min at 0 to 5°C, and the precipitate of sodium chloride was removed by filtration and washed with methanol. The solution was concentrated in vacuo and diluted with 125 ml of methylene dichloride. To this solution was added portionwise 30 ml of acetic anhydride. A vigorous reaction occurred with a white solid separating. The reaction mixture was refluxed for 1 hr, cooled, and filtered. The yield was 29.8 g (93.2%), mp 178 to 180°C. Recrystallization from methanol did not raise the melting point.

Anal. Calc'd for $\text{C}_{10}\text{H}_{20}\text{N}_6\text{O}_6$: %C, 37.49; %H, 6.29; %N, 26.24

Found: %C, 38.25; %H, 6.75; %N, 25.80

(2) $\text{N,N}'\text{-Diacetyl-3,6-dinitraza-1,8-octane diamine}$ was added to a mixture of acetic anhydride and 100% nitric acid at 5 to 10°C. When the acid solution was poured on ice, only water-soluble products were obtained.

5. Conversion of $\text{N,N-bis(2,2-Dinitropropyl)aminomethyl Isocyanate}$ to $\text{bis(2,2-Dinitropropyl) Nitrosoamine}$

a. Discussion

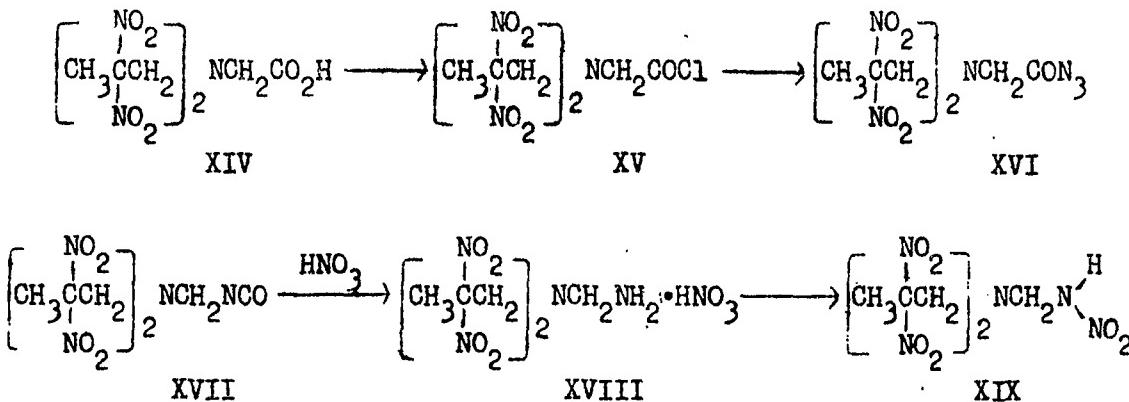
(1) In continuing the study of the preparation of primary nitramines it was of interest to convert $\text{N,N-bis(2,2-dinitropropyl) glycine}$ (XIV)* to $\text{N,N-bis(2,2-dinitropropyl)-N'-nitromethylene diamine}$ (XIX):

* Aerojet Report No. 622, p. 85.

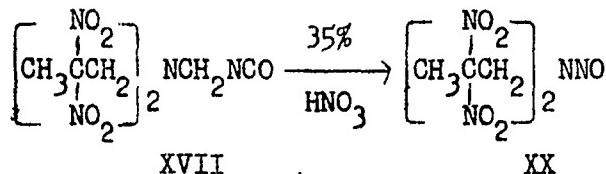
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II Technical Progress, E (cont.)

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XIV was converted to the corresponding isocyanate via the acid chloride (XV) and azide (XVI). When N,N-bis(2,2-dinitropropyl)aminomethyl isocyanate (XVII) was treated with 35% nitric acid an extremely vigorous reaction occurred after a short time, and bis(2,2-dinitropropyl) nitrosoamine (XX) was obtained instead of the expected N,N-bis(2,2-dinitropropyl) methylene diamine nitrate (XVIII).



These results are in accord with earlier work* wherein XX was prepared by treating XIV with a mixture of acetic anhydride and 100% nitric acid, thus indicating the instability of XIV and XVII toward nitric acid.

b. Experimental

(1) Preparation of N,N-bis(2,2-Dinitropropyl)-glycyl Chloride

A mixture of 78 g (0.23 mole) of N,N-bis(2,2-dinitropropyl) glycine and 190 ml of thionyl chloride was refluxed overnight. The solution was evaporated to dryness in vacuo, leaving a brown solid. Two recrystallizations from chloroform gave white crystals; the yield was 41 g (50%), mp 64 to 65°C.

Anal. Calc'd for C₈H₁₂N₂O₅Cl: %C, 26.86; %H, 3.38; %N, 19.14
%Cl, 9.91

Found: %C, 27.48; %H, 3.68; %N, 19.27;
%Cl, 9.70

* Aerojet Report No. 622, p. 84.

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II Technical Progress, E (cont.)

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(2) Preparation of N,N-bis(2,2-Dinitropropyl) Nitrosoamine

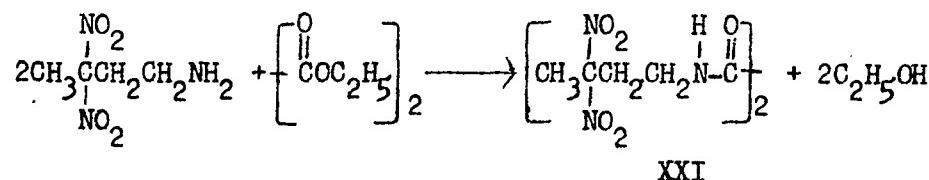
A solution of 15.2 g (0.23 mole) of sodium azide in 150 ml of water was placed in a 500-ml three-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel. The flask was cooled in an ice bath, and a solution of 31.0 g (0.087 mole) of N,N-bis (2,2-dinitropropyl) glycyl chloride in 130 ml of acetone was added drop-wise, keeping the temperature below 10°C. The reaction mixture was extracted with chloroform, and the extracts were combined, washed with water, and dried over sodium sulfate at 10°C. The solution was heated to reflux and the azide was decomposed while the chloroform was being distilled to remove the last traces of water. The isocyanate solution was cooled to room temperature, and 100 ml of 35% nitric acid was added. The reaction mixture was warmed on the steam bath, and after about 30 min an extremely vigorous reaction occurred with evolution of gas. It was necessary to cool the reaction mixture in an ice bath until the reaction subsided. A white solid separated from the reaction mixture, and was collected and dried. The yield was 16.0 g (59.5%), mp 129 to 130°C. A sample recrystallized from ethylene dichloride melted at 132 to 133°C, gave a positive Lieberman test for a nitroso group, and gave no depression in melting point when mixed with an authentic sample of bis(2,2-dinitropropyl) nitrosamine.

F. PREPARATION OF ALIPHATIC POLYNITRONITRAMIDES

1. Preparation of N,N'-bis(3,3-Dinitrobutyl) Oxamide

a. Discussion

The preparation of N,N'-bis(3,3,3-trinitropropyl) oxamide from 3,3,3-trinitropropyl amine and oxalyl chloride has been previously reported.* This reaction has now been extended to the preparation of N,N'-bis(3,3-dinitrobutyl) oxamide (XXI) by the condensation of 3,3-dinitrobutyl amine with ethyl oxalate.



XXI is a white crystalline solid, mp 190 to 191°C.

* Aerojet Report No. 711, p.8.

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b. Experimental

A 35.0-g (0.175 mole) quantity of 3,3-dinitrobutyl amine hydrochloride, 200 ml of water, and 175 ml of chloroform was placed in a one-liter three-necked flask, fitted with a mechanical stirrer, thermometer, and dropping funnel. At room temperature a solution of 7.1 g (0.175 mole) of sodium hydroxide in 150 ml of water was added dropwise. The chloroform layer was separated, and the aqueous solution was extracted with 2 50-ml portions of chloroform. The chloroform extracts were combined and dried over sodium sulfate for 30 min. The solution was filtered into a one-liter round-bottom flask, 12.8 g (0.088 mole) of ethyl oxalate was added, and the reaction mixture was refluxed for 2 hr. The solution was concentrated in vacuo, leaving a cream-colored solid. Recrystallization from ethylene dichloride gave 13.0 g (39%) of white crystals, mp 190 to 191°C. A second recrystallization did not raise the melting point.

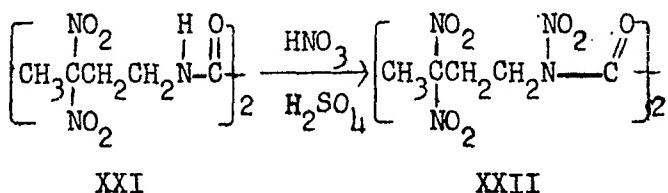
Anal. Calc'd for $C_{10}H_{16}N_6O_{10}$: %C, 31.58; %H, 4.24; %N, 22.10

Found: %C, 31.75; %H, 4.43; %N, 22.65

2. Preparation of N,N'-Dinitro-N,N'-bis(3,3-dinitrobutyl) Oxamide

a. Discussion

The nitration of N,N'-bis(3,3-dinitrobutyl) oxamide (XXI) with a mixture of 100% nitric acid and concentrated sulfuric acid at 55°C gave N,N'-dinitro-N,N'-bis(3,3-dinitrobutyl) oxamide (XXII). XXII is a white crystalline solid, existing in two polymorphic forms, mp 142 to 143°C and 148 to 149°C, with a calculated lead-block value of 118 and a calculated ballistic-mortar value of 129.



b. Experimental

A 15.0-g (0.395 mole) quantity of N,N'-bis(3,3-dinitrobutyl) oxamide was added to a mixture of 175 ml of 100% nitric acid and 150 ml of concentrated sulfuric acid. The reaction mixture was heated to 55°C for 30 min, cooled, and poured on ice. The white solid was collected, washed with water, and dried. The yield was 17.6 g (94.8%), mp 136 to 137°C. Recrystallization from ethylene dichloride gave a compound melting at 142 to 143°C and 148 to 149°C.

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Anal. Calc'd for $C_{10}H_{14}N_8O_{14}$: %C, 25.54; %H, 3.00; %N, 23.83

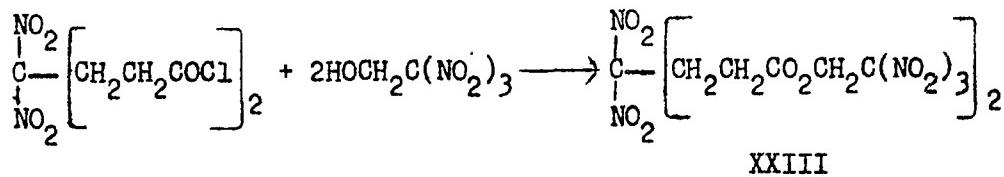
Found: %C, 25.84; %H, 3.26; %N, 24.37

G. PREPARATION OF ALIPHATIC POLYNITROESTERS

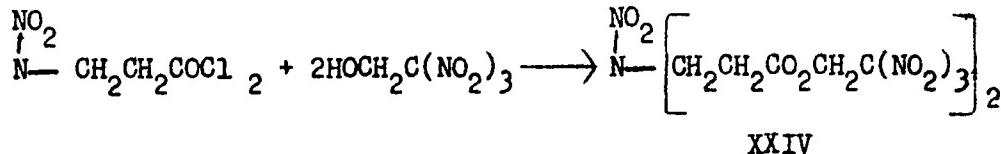
1. Discussion

a. An improved method for the preparation of esters of 2,2,2-trinitroethanol has been described by the Naval Ordnance Laboratory.* This method involves the use of small amounts of anhydrous aluminum chloride with an acid chloride and 2,2,2-trinitroethanol, thus permitting the reaction to proceed rapidly under mild conditions either with or without a solvent. Some of the 2,2,2-trinitroethanol esters are of considerable interest as new high explosives because of their high oxygen content and good thermal stability. In addition, certain of these esters have polymerization properties which are being investigated for propellant compositions and others may be useful as explosive waxes. For these reasons it was of interest to convert some of the acid chlorides prepared on the nitro-polymer and explosives programs to the corresponding 2,2,2-trinitroethyl esters. Accordingly, the following esters were prepared:

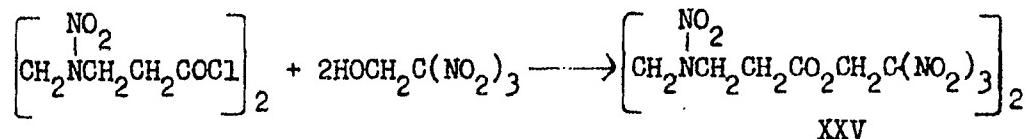
(1) bis(2,2,2-Trinitroethyl)-4,4-dinitro-heptanedioate (XXIII), mp 170 to 171°C:



(2) bis(2,2,2-Trinitroethyl)-4-nitro-az-heptanedioate (XXIV), mp 110 to 111°C:



(3) bis(2,2,2-Trinitroethyl)-4,7-dinitro-az-decanedioate (XXV), mp 126 to 128°C:



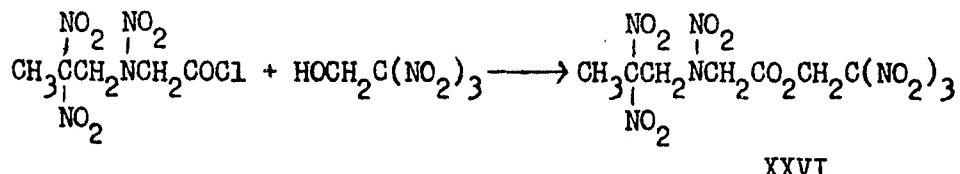
* NAVORD Report 2245.

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(4) 2,2,2-Trinitroethyl-3,5,5-trinitro-3-aza-hexanoate (XXVI), mp 121 to 121.5°C:



b. 4,4,6,8,8-Pantanitro-6-aza-undecanedioyl chloride failed to react with 2,2,2-trinitroethanol. N-Nitro-N-trinitroethyl aspartyl chloride and N-nitro-N-trinitroethyl glutamyl chloride gave oils when treated with 2,2,2-trinitroethanol, indicating that decomposition had occurred.

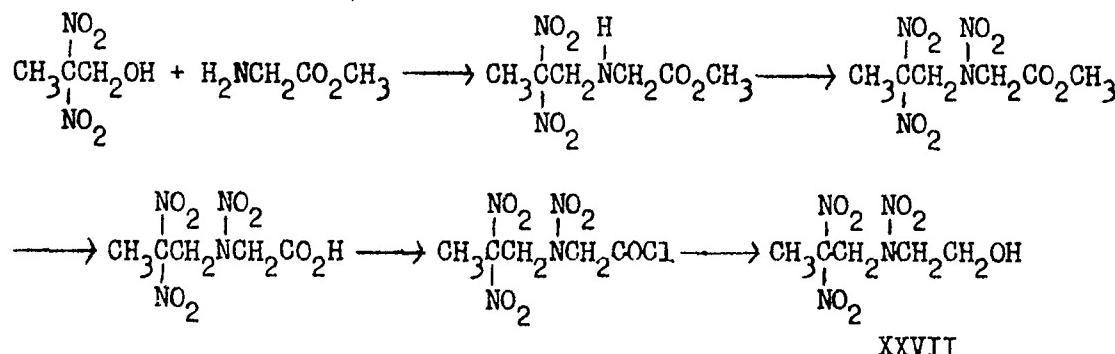
2. Experimental

The general procedure used* is illustrated in the following example. A solution of 3.62 g (0.02 mole) of 2,2,2-trinitroethanol in 50 ml of dry ethylene dichloride was placed in a 100-ml round-bottom flask, fitted with a condenser and drying tube. Anhydrous aluminum chloride, 0.27 g (0.002 mole), was added, and the solution turned yellow. To the solution was added 3.31 g (0.01 mole) of 4,7-dinitraza-decanedioyl chloride. The reaction mixture was refluxed until evolution of hydrogen chloride gas had ceased. The black mixture was cooled and filtered, and the solid was washed with cold dilute hydrochloric acid, and water, then dried and recrystallized from ethylene dichloride, using charcoal, to give 5.3 g (85.5%) of white crystals, mp 126 to 128°C. All the results are summarized in Table III.

H. PREPARATION OF 1-NITRATO-3,5,5-TRINITRO-3-AZA-HEXANE

1. Discussion

The preparation of 3,5,5-trinitro-3-aza-1-hexanol (XXVII) has been previously reported.**



* NAVORD Report 2245.

** Aerojet Reports No. 711 and 712.

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TABLE III

ALIPHATIC POLYNITROESTERS

Compound	Yield, %	mp °C	Recryst. Solvent	Formula	Analyses			
					Calc'd	Found	C	H
					C	H	N	
$\text{NO}_2 \left[\text{CH}_2 \text{CH}_2 \text{CO}_2 \text{CH}_2 \text{C}(\text{NO}_2)_3 \right]_2 \text{NO}_2$	47.0	170-171	Ethylene dichloride	$\text{C}_{11}\text{H}_{12}\text{N}_8\text{O}_{20}$	22.93	2.10	19.45	23.18 2.09 18.74
$\text{NO}_2 \left[\text{CH}_2 \text{CH}_2 \text{CO}_2 \text{CH}_2 \text{C}(\text{NO}_2)_3 \right]_2$	35.9	110-111	Chloroform	$\text{C}_{10}\text{H}_{12}\text{N}_8\text{O}_{18}$	22.56	2.27	21.05	22.58 2.26 20.93
$\text{NO}_2 \text{CH}_2 \text{CH}_2 \text{CO}_2 \text{CH}_2 \text{C}(\text{NO}_2)_3$	85.5	126-128	Ethylene dichloride	$\text{C}_{12}\text{H}_{16}\text{N}_{10}\text{O}_{20}$	23.23	2.60	22.58	23.49 2.90 23.10
$\text{CH}_3 \text{C}(\text{NO}_2)_2 \text{CH}_2 \text{CO}_2 \text{CH}_2 \text{C}(\text{NO}_2)_3$	50.3	121-121.5	Ethylene dichloride	$\text{C}_7\text{H}_9\text{N}_7\text{O}_{14}$	20.25	2.19	23.62	20.37 2.09 23.70
			NO_2					

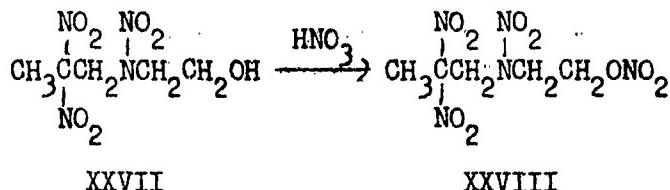
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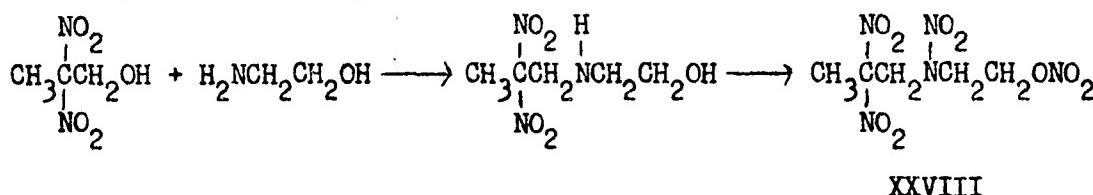
II Technical Progress, H (cont.)

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In continuation of the work on the preparation of aliphatic polynitronitrates,* XXVII was treated with 100% nitric acid to give 1-nitro-3,5,5-trinitro-3-aza-hexane (XXVIII).



XXVIII is a white crystalline solid, mp 117 to 118°C, with a calculated lead-block value of 142 and a calculated ballistic-mortar value of 146. Compound XXVIII was prepared independently on the nitropolymer program** in the following manner:

2. Experimental

Thirty ml of 100% nitric acid was cooled to 5°C and 3.0 g (0.012 mole) of 3,5,5-trinitro-3-aza-1-hexanol† was added. The solution was stirred for 15 min at 5°C and poured on ice. The white solid was collected, washed with water, and dried; the yield was 3.3 g (92.7%), mp 75 to 98°C. Two recrystallizations from methanol raised the melting point to 117 to 118°C.

Anal. Calc'd for C₅H₉N₅O₉: %C, 21.21; %H, 3.20; %N, 24.74

Found: %C, 21.74; %H, 3.51; %N, 25.31

There was no depression in melting point when this sample was mixed with the 116 to 117°C-mp compound obtained from the nitration of the condensation product of 2,2-dinitropropanol and ethanalamine.

* Aerojet Report No. 737, p. 10.

** Aerojet Report No. 772, in press.

† Aerojet Report No. 712, p. 43.

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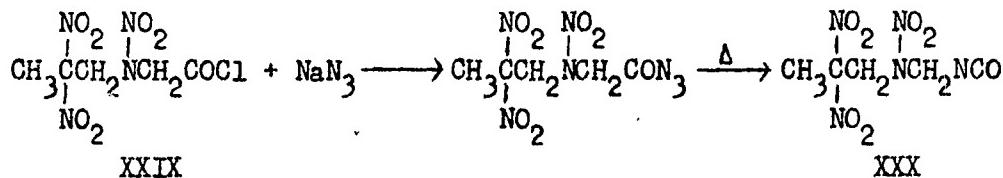
II Technical Progress (cont.)

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I. PREPARATION OF 2,4,4-TRINITRO-2-AZA-AMYL ISOCYANATE

1. Discussion

2,4,4-Trinitro-2-aza-amyl isocyanate (XXX) has been prepared in situ from 3,5,5-trinitro-3-aza-hexanoyl chloride (XXIX) and used directly for the synthesis of high explosives:^{*}



XXX has now been isolated, purified, and identified. The compound, mp 102 to 103°C, explodes violently on standing at room temperature. It can be stored safely at -20°C, but for synthetic purposes the isocyanate is best used in solution without being isolated.

2. Experimental

A solution of 35.1 g (0.54 mole) of sodium azide in 250 ml of water was placed in a one-liter three-necked flask, fitted with a thermometer, mechanical stirrer, and dropping funnel. The solution was cooled to 5°C with an ice bath, and a solution of 73 g (0.27 mole) of 3,5,5-trinitro-3-aza-hexanoyl chloride in 200 ml of acetone was added dropwise, keeping the temperature below 10°C. The reaction mixture was stirred for 30 min and extracted with three 150-ml portions of chloroform. The extracts were combined, washed with two 100-ml portions of water, two 100-ml portions of 10% sodium bisulfate, and two 100-ml portions of water. The chloroform solution was dried for 30 min over sodium sulfate and placed in a one-liter round-bottom flask arranged for distillation. About 100 ml of chloroform was stripped off with the water aspirator to remove the last traces of water. The temperature was raised to 60 to 65°C with the steam bath, and the azide was decomposed at atmospheric pressure. Dry chloroform was added periodically to keep the volume constant. After the nitrogen evolution had ceased the solution was cooled to -10°C, causing a white solid to separate. The product was collected and dried. The yield of 2,4,4-trinitro-2-aza-amyl isocyanate was 48.1 g (72%), mp 97 to 100°C. Recrystallization from ethylene dichloride raised the melting point to 102 to 103°C. The compound explodes violently on standing at room temperature but can be safely stored at -20°C. For synthetic purposes it is best used in solution without being isolated.

Anal. Calc'd for C₅H₇N₅O₇: %C, 24.10; %H, 2.83; %N, 28.11

Found: %C, 24.31; %H, 3.11; %N, 28.05

* Aerojet Report No. 711, p. 11.

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II Technical Progress (cont.)

Report No. 770

J. PREPARATION OF SAMPLES FOR EVALUATION BY THE NAVAL ORDNANCE LABORATORY

1. The following samples were prepared and submitted to the Naval Ordnance Laboratory for evaluation:

- a. 2,2,5,9,9,13,16,16-Octanitro-5,13-diaza-6,12-diketo-7,11-dioxa-heptadecane
- b. 1,1,1,5,8,11,14,18,18,18-Decanitro-3,16-dioxa-4,15-diketo-5,8,11,14-tetraza-octadecane
- c. 1,1,1,5-Tetranitro-3-oxa-4-keto-5-aza-hexane
- d. 1,1,1,5-Tetranitro-3-oxa-4-keto-5-aza-heptane
- e. 3,5,5-Trinitro-3-aza-hexyl Nitrate
- f. 1,1,1,3,6,9,12,14,14,14-Decanitro-3,6,9,12-tetraza-tetradecane
- g. 1,1,1,3,6,9,11,11,11-Nonanitro-3,6,9-triazaundecane
- h. N,N'-Dinitro-N,N'-bis(3,3-dinitrobutyl) Oxamide
- i. bis(2,2,2-Trinitroethyl)-4,4-dinitroheptanedioate
- j. bis(2,2,2-Trinitroethyl)-4-nitraheptanedioate
- k. bis(2,2,2-Trinitroethyl)-4,7-dinitraza-decanedioate
- l. 2,2,2-Trinitroethyl-3,5,5-trinitro-3-azahexanoate

2. The results of the preliminary tests by the Naval Ordnance Laboratory on the compounds submitted are summarized in Table IV. The noteworthy feature is the excellent thermal stability exhibited by compounds A53-15 and A53-31, both of which are aliphatic polynitronitrates.

3. SPIA data sheets, which are presented in the appendix, have been completed on the following compounds:

- a. 3,3-Dinitrobutyl Ammonium Nitrate
- b. 3,3-Dinitro-1,5-pentane Diammonium Dinitrate
- c. 3-Nitraza-1,5-pentane Diammonium Dinitrate
- d. 3,6-Dinitraza-1,8-octane Diammonium Dinitrate
- e. 3,3,5,7,7-Pantanitro-5-aza-1,9-nonane Diammonium Dinitrate
- f. 1,7-Dinitrato-4,4-dinitroheptane

Dinitrate

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II Technical Progress, J (cont.)

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TABLE IV
NOL PRELIMINARY TESTS ON HIGH EXPLOSIVES

Compound	mp °C	I.S. cm/2.5 kg in.-3	Cryst. Dens. g in.-3	Ignition Temp. °C	Thermal Stability (Gas Vol/g at 100°C)	Calc. Block Value in 48 hr (TNT = 100)	Lead- Ballistic- Mortar Value (TNT = 100)
$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2\text{CCH}_2\text{CH}_2\text{CH}_2\text{ONO}_2 \end{array} \right]_2$	73-75	34 (Class 4)	1.63	217	More than 30 cc/g in 12 hr (Class IV)	126	137
A53-13							
$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2\text{CCH}_2\text{CH}_2\text{CH}_2\text{ONO}_2 \end{array} \right]_2$	100-101	28 (Class 4)	1.70	172	More than 30 cc/g in 2 hr (Class IV)	138	144
A53-14							
$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2\text{CCH}_2\text{CH}_2\text{CH}_2\text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2$	133-135	86 (Class 2)	1.67	227	Good (Class I)	135	142
A53-15							

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II Technical Progress, J (cont.)

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TABLE IV (cont.)

Compound	mp °C	I.S. cm/2.5 kg	Dens. g in.-3	Cryst. Temp. °C	Ignition in 48 hr	Thermal Stability (Gas Vol/g at 100°C)	Calc. Lead- Ballistic- Value (TNT = 100)	Calc. Mortar Value (TNT = 100)
$\text{NO}_2 \text{C}(\text{CH}_2\text{NH}_2\cdot\text{HNO}_3)\text{NO}_2$	131-133	52	1.62	288	0.2 (Class I)	112	116	
A53-22								
$\text{NO}_2 \left[\text{CH}_2\text{CH}_2\text{NH}_2\cdot\text{HNO}_3 \right]_2 \text{NO}_2$	151-153	60	1.64	280	2.0 (Class I)	120	130	
A53-23								
$\text{NO}_2 \left[\text{CH}_2\text{CH}_2\text{NH}_2\cdot\text{HNO}_3 \right]_2 \text{NO}_2$	153-155	61	1.57	281	0.0 (Class I)	112	126	
A53-24								
$\left[\text{NO}_2 \text{CH}_2\text{NH}_2\cdot\text{HNO}_3 \right]_2 \text{NO}_2$	185-190	58	1.60	317	0.0 (Class I)	109	125	
A53-25								

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II Technical Progress, J (cont.)

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TABLE IV (cont.)

Compound	mp °C	I.S. cm/2.5 kg	Cryst. Dens. g in.-3	Ignition Temp. °C	Thermal Stability (Gas Vol/g at 100°C) in 48 hr	Calc. Ballistic- Mortar Value (TNT = 100)
A53-26	$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2 \text{CCH}_2 \text{CH}_2 \text{NH}_2 \cdot \text{HNO}_3 \end{array} \right]_2$ NO_2	175-180 (Class I)	37	1.86	197 Over 30 (Class IV)	135 144
A53-27	$\text{NO}_2 \left[\begin{array}{c} \text{ONO} \\ \\ \text{CH}_2 \text{C}(\text{CNCH}_2 \text{CH}_2 \text{CCH}_3) \text{NO}_2 \end{array} \right]_2$ NO_2	117-118 (Class 2)	137	1.66	232 1.1 (Class I)	119 128
A53-28	$\left[\begin{array}{c} \text{NO}_2 \\ \\ \text{CH}_2 \text{NCH}_2 \text{CH}_2 \text{NCO}_2 \text{CH}_2 \text{C}(\text{NO}_2)_2 \end{array} \right]_2$	127-128 (Class 4-5)	20	1.78	213 22.5 (Class IV)	112 141

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TABLE IV (cont.)

Compound	mp °C	I.S. cm ² /2.5 kg	Cryst. Dens. g/in.-3	Ignition Temp. °C.	Thermal Stability (Gas Vol/g at 100°C) in 48 hr	Calc. Mortar Value (TNT = 100)	Lead-Ballistic- Mortar Value (TNT = 100) (TNT = 100)	Calc.
NO_2 $\text{CH}_3\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$	39-40	17 (Class 4-5)	1.72	231	13.1 (Class IV)	168	137	
A53-29								
NO_2 $\text{C}_2\text{H}_5\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$	50-51	19 (Class 4-5)	1.69	235	28.4 (Class IV)	146	146	136
A53-30								
NO_2 NO_2 $\text{CH}_3\text{CCH}_2\text{NCH}_2\text{CH}_2\text{ONO}_2$	116-117	21 (Class 4-5)	1.73	---	1.1 (Class I) 1.5 in 120 hr (Class I)	142	146	
A53-31								

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II Technical Progress, J (cont.)

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- g. 1-Nitrato-3,5,5,-trinitro-3-aza-hexane
- h. 2,2,6,9,9-Pantanitro-4-oxa-5-keto-6-aza-decane
- i. 2,2,6,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane
- j. 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane
- k. 1,1,1,5,8,11,15,15,15-Nonanitro-3,13-dioxa-4,12-diketo-5,8,11-triaza-pentadecane
- l. 1,1,1,5,8,8,10,12,12,15,19,19,19,19-Tridecanitro-3,17-dioxa-4,16-diketo-5,10,15-triaza-nonadecane
- m. 2,2,5,9,9,13,16,16-Octanitro-5,13-diaza-6,12-diketo-7,11-dioxa-heptadecane
- n. 1,1,1,5,8,11,14,18,18,18-Decanitro-3,16-dioxa-4,15-diketo-5,8,11,14-tetraza-octadecane
- o. 1,1,1,5-Tetranitro-3-oxa-4-keto-5-aza-heptane
- p. N,N'-Dinitro-N,N'-bis(3,3-dinitrobutyl) Oxamide

K. HEATS OF FORMATION AND OXYGEN CONTENTS OF EXPLOSIVE COMPOUNDS

1. It has been the practice on this program to assess the explosive power of new compounds in terms of the ballistic-mortar and lead-block values. However, there are other functions which may also be useful for this purpose, and are favored by other investigators. Two of these are:

a. The heat of formation at 25°C and 1 atm, following the usual thermochemical conventions. (With negligible error, this is the heat of formation at bomb temperature and pressure.)

b. The oxygen content, expressed as gram atoms of oxygen per milliliter of compound.

2. A compilation of these values for compounds for which SPIA data sheets were prepared on this program is given in Table V. Compounds are listed in order of decreasing heats of formation. Heats of formation were calculated from experimental heats of combustion, and oxygen contents from experimental densities in all cases. These values may be compared with the following for standard explosives:

	<u>Heat of Formation, kcal/mole</u>	<u>Oxygen Content, gram-atoms/mole</u>
RDX	+20	0.0491
Nitroglycerine	-85	0.0630
TNT	-15	0.0437

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II Technical Progress, K (cont.)

TABLE V

SUMMARY OF HEATS OF FORMATION AND
OXYGEN CONTENTS OF EXPLOSIVE COMPOUNDS

	<u>Heat of Formation, kcal/mole</u>	<u>Oxygen Content, gram-atoms/ml</u>		<u>Heat of Formation, kcal/mole</u>	<u>Oxygen Content, gram-atoms/ml</u>
<chem>CH2[N+](=O)[C-]2CC(C(=O)N2)C</chem>	-18	0.0617	<chem>[NO2]3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-90	0.0610
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-19	0.0531	<chem>(NO2)3CCH2CH2NH2+HNO3</chem>	-95	0.0621
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-20	0.0508	<chem>O=C([NHCH2CH2C([NO2]2)]2)</chem>	-98	0.0540
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-28	0.0637	<chem>C([NO2]2)-[CH2CH2N[NO2]CH2C([NO2]2)CH3]2</chem>	-104	0.0500
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-31	0.0591	<chem>CH3CH2N[NO2]CO2CH2C([NO2]2)CH3</chem>	-106	0.0570
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-33	0.0590	<chem>(NO2)3CCH2CH2N[NO2]CO2CH2C([NO2]2)CH3</chem>	-111	--
<chem>(NO2)3CCH2CH2N[NO2]</chem>	-36	0.0611	<chem>CH3C([NO2]2)CH2N[NO2]CH2N[NO2]CO2CH2C([NO2]2)CH3</chem>	-118	0.0603
<chem>CH3C([NO2]2)CH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-40	0.0563	<chem>CH3C([NO2]2)CH2CH2NH2+HNO3</chem>	-121	0.0499
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-42	0.0630	<chem>N[NO2]-[CH2C([NO2]2)CH2CH2N[NO2]CO2CH2C([NO2]2)CH3]2</chem>	-130	0.0595
			<chem>CH3C([NO2]2)CH2CH2N[NO2]CO2CH2C([NO2]2)CH3</chem>	-132	0.0585
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-44	0.0583	<chem>[NO2]3C([CH2ONO2]2)2</chem>	-134	0.0701
<chem>C([NO2]2)-[CH2N[NO2]CH2CH2C([NO2]2)]2</chem>	-50	0.0595	<chem>[NO2]3CCH2CH2NH-C(=O)</chem>	-145	0.0536
<chem>C([NO2]2)-[CH2CH2NH[NO2]]2</chem>	-54	0.0474	<chem>CH3C([NO2]2)CH2CH2N[NO2]C</chem>	-145	--
<chem>(NO2)2C([NO2]2)CH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-60	0.0510	<chem>CH3C([NO2]2)CH2CH2N[NO2]CO2CH2C([NO2]2)CH3</chem>	-147	0.0504
<chem>C([NO2]2)-[CH2CH2N[NO2]CH2C([NO2]2)CH3]2</chem>	-60	0.0605	<chem>N[NO2]-[CH2CH2N[NO2]CO2CH2C([NO2]2)CH3]2</chem>	-159	0.0596
<chem>C([NO2]2)-[CH2N[NO2]CH2CH2C([NO2]2)CH3]2</chem>	-62	0.0491	<chem>[CH2N[NO2]CH2CH2NH2+HNO3]2</chem>	-159	0.0460
<chem>N[NO2]-[CH2C([NO2]2)CH3]2</chem>	-65	0.0530	<chem>C([NO2]2)-[CH2O2CH2N[NO2]CH2CH2C([NO2]2)CH3]2</chem>	-167	0.0600
<chem>CH3C([NO2]2)CH2N[NO2]CO2CH2ONO2</chem>	-73	0.0550	<chem>CH2-[N[NO2]-CO2CH2C([NO2]2)]2</chem>	-168	0.0688
<chem>N[NO2]-[CH2C([NO2]2)CH3]2</chem>	-85	0.0482	<chem>N[NO2]-[CH2CH2NH2+HNO3]2</chem>	-170	0.0459
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>	-86	0.0572	<chem>C([NO2]2)-[CH2CH2N[NO2]C]2</chem>	-188	0.0516
<chem>(NO2)3CCH2CH2N[NO2]CH2C([NO2]2)CH3</chem>			<chem>[CH2N[NO2]CH2CH2N[NO2]CO2CH2C([NO2]2)CH3]2</chem>	-190	0.0578
			<chem>(NO2)3CCH2CH2N[NO2]CO2CH2C([NO2]2)CH3</chem>	-192	0.0619
			<chem>N[NO2]-[CH2C([NO2]2)CH2CH2NH2+HNO3]2</chem>	-209	--
			<chem>C([NO2]2)-[CH2CH2N[NO2]CO2CH2C([NO2]2)CH3]2</chem>	-215	0.0606
			<chem>(NO2)2C([CH2O2CN[NO2]CH2CH2C([NO2]2)CH3]2)</chem>	-244	0.0524

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III. DESENSITIZATION OF RDX

The possibility of desensitizing RDX by coating with surface-active compounds containing nitro groups has been previously discussed.* Following this idea, 3,3,3-trinitro propyl isocyanate and glycerol mono-oleate were reacted in varying ratios with the objective of substituting the glycerol hydroxyl groups to various extents. Three products were obtained, corresponding to ratios of carbamate to glycerol mono-oleate of 1-1/4 to 1, 1-1/2 to 1, and 2 to 1. Each of these was a waxy solid, melting slightly above room temperature.

Each of the three products was applied in 10% ratio to RDX by the method for preparing Composition A.** The desensitized specimens obtained were tested with the Bureau of Mines impact tester, with the following results:

	<u>Impact Stability, cm/2 kg</u>	
	<u>On bare anvil</u>	<u>On 5/0 sandpaper</u>
1-1/4 to 1 Ratio product	100	20-25
1-1/2 to 1 Ratio product	100	20-25
2 to 1 Ratio product	100	20-25
Composition A-3	100	45-50
RDX	40	10-15

This degree of desensitization compares well with that obtained using nitropolymers, but its inferiority to the value for Composition A is disappointing.

In another desensitization experiment, 10% 2,2-dinitro propanediol was applied to RDX by the same procedure, using no surface-active agent. The impact stability of the product was 30 to 35 cm/2kg on the bare anvil.

These lines of investigation were discontinued because of the termination of work with RDX under the present contract.

*Aerojet Report No. 737, p. 23.

**Aerojet Report No. 682, p. 21.

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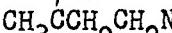
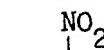
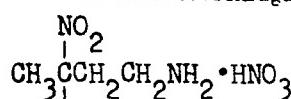
Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 3,3-Dinitrobutyl 1-ammonium

Name nitrateEmpirical formula C₄H₁₀N₄O₇

Structure:(configuration)

Preparation reaction(s): NO₂

Information submitted by:

Activity Aerojet-General CorporationPerson M.B. Frankel and L.T. CarletonDate 1 November 1953

1. Quantitative analysis(% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	21.24	4.46		24.78		
By determination	21.39	4.63		25.19		

2. Burning properties:(compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD 3401 p.8	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	48 hr at 100°C
c. Vacuum Stability	OSRD 3401 p.10	
d. Temperature of Explosion	OSRD 3401 p.6	
e. Temperature of Ignition	OSRD 3401 p.6	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, N.C., etc.)

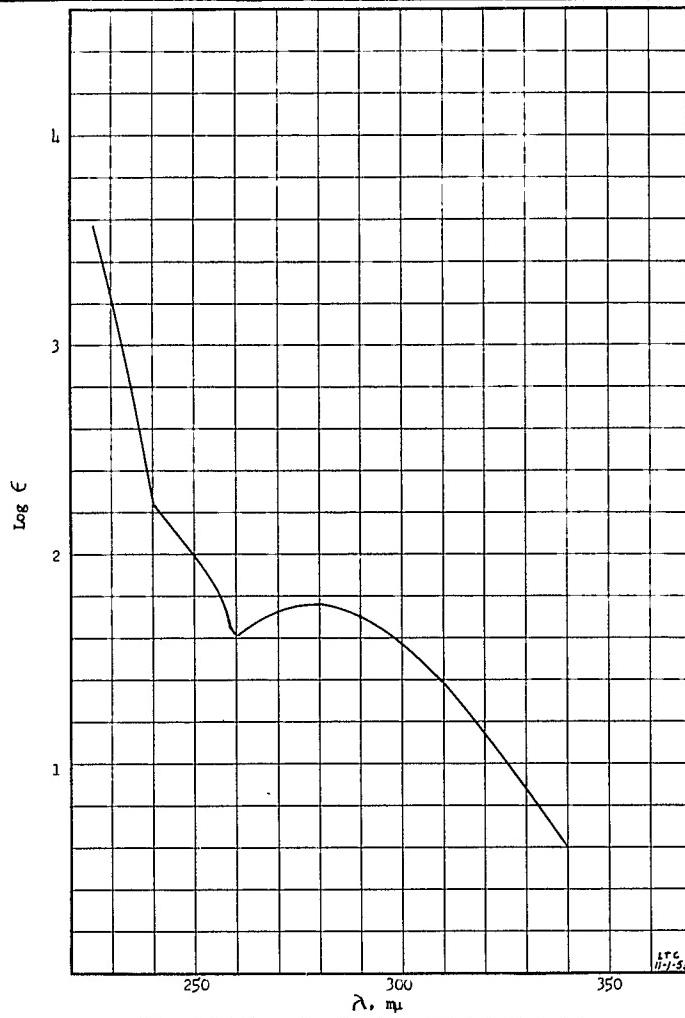
New Compound test results

a. Tetryl, 32 cm/2.5 kg	52 cm/2.5 kg
b.	
c.	0.2 cc/g
d.	
e.	288°C
f.	
g.	
h.	

4. Heat of formation: (ΔH) + -121 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2629	cal/gm	2725
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)		flat, white crystals	
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method) 1.62 gm/cm ³ . (NOL) (Micro or other method)		gm/cm ³ .	
11. Index of refraction: (n _D ^{25°C.})		12. Color	White
14. pH at 25°C. 1.1 solvent and concentrations used. pH indicator paper or Beckman pH meter.)		13. Odor	None

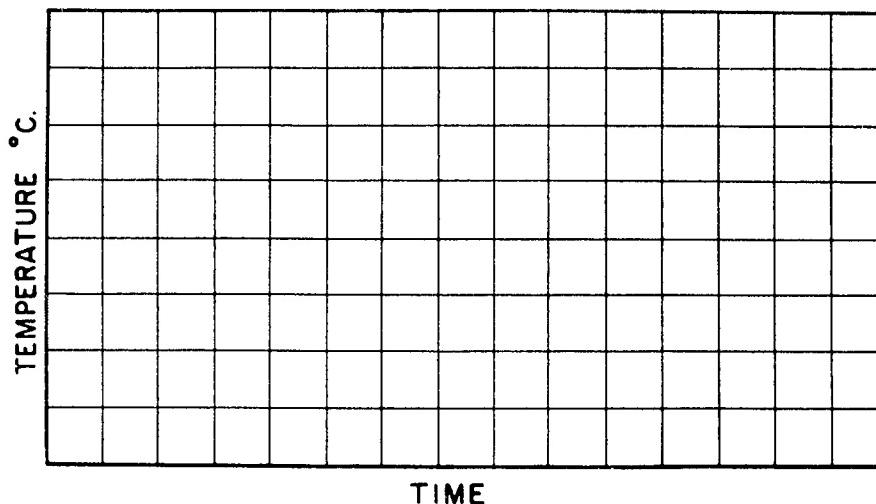


Ultraviolet Absorption Spectrum of 3,3-Dinitrobutyl
1-Ammonium Nitrate in Methanol

20. Melting point: 131-133 °C.

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21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



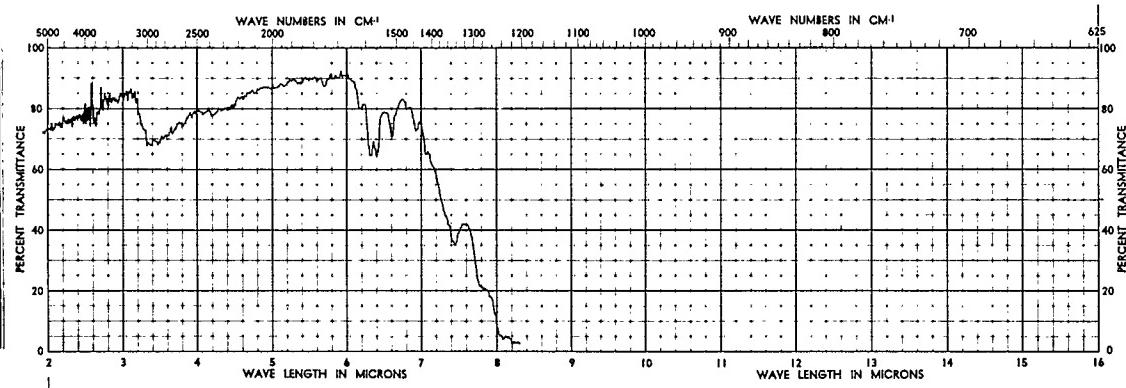
22. Solubility of new compound:

19 g/100 ml H₂O at 25°C. g/100 ml H₂O at _____ °C.
 5 g/100 ml acetone at 25 °C.
 < 0.1 g/100 ml toluene at 25 °C.

(name material used as solvent)

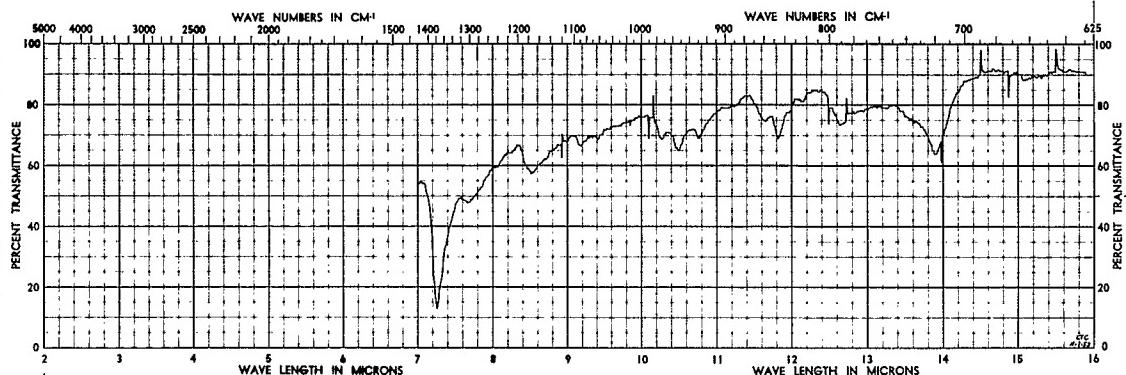
I. R. SPECTROPHOTOMETER
NaCl PRISM
NO. DATE INDEX
10-8-53 SAMPLE
3,3-Dinitrobutyl 1-Amonium
nitrate
Suspended in Fluorolube

FROM Time = 12 min
SAMP. CELL MM CMS.
REF. CELL MM CMS.
CHEM. MG. MG.
SOLV.
VOL. C.C. % C.C. %
F.S. SOLID C. GAS MM
BAIRD ASSOCIATES, INC.
CAMBRIDGE, MASS., U.S.A.
10214



I. R. SPECTROPHOTOMETER
NaCl PRISM
NO. DATE INDEX
10-8-53 SAMPLE
3,3-Dinitrobutyl 1-Amonium
nitrate
Suspended in white mineral oil

FROM Time = 12 min
SAMP. CELL MM CMS.
REF. CELL Salt Block CMS.
CHEM. MG. MG.
SOLV.
VOL. C.C. % C.C. %
F.S. SOLID C. GAS MM
BAIRD ASSOCIATES, INC.
CAMBRIDGE, MASS., U.S.A.
10214



CONFIDENTIAL

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "G" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thickol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? research quantities _____
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existance, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____
Calculated lead-block value = 112) Method of Aerojet Report No. 512, p.8.
Calculated Ballistic-Mortar Value = 116)
- _____

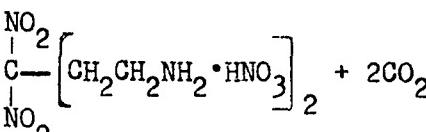
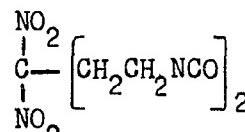
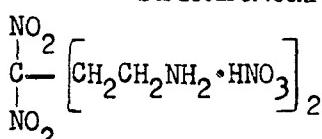
CONFIDENTIAL

Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 3,3-Dinitro 1,5-pentane

Name diammonium dinitrate
Empirical formula C₅H₁₁N₆O₁₀
Structure: (configuration)



Preparation reaction(s):

Information submitted by:
Activity Aerojet-General Corporation
Person M.B.Frankel and L.T.Carleton
Date 1 November 1953

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	18.87	4.43		26.41		
By determination	19.07	4.70		26.22		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method
a. Impact Sensitivity	NOISEMEX
b. Thermal Stability	OSRD 3401 p.8
c. Vacuum Stability	NOISEMEX 3401 p.8
d. Temperature of Explosion	OSRD 3401 p.8
e. Temperature of Ignition	NOISEMEX 3401 p.8
f.	
g.	
h.	

2.5-kg weight

48 hr at 100°C

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, N.C., etc.)

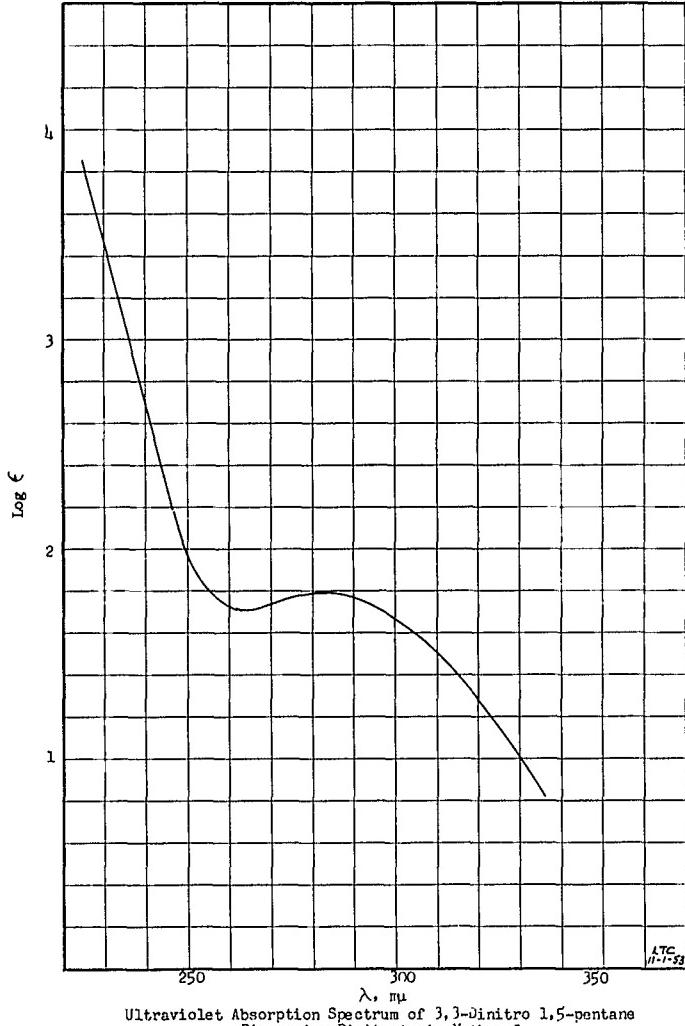
New Compound test results

a. Tetryl, 32 cm/2.5 kg	60 cm/2.5 kg
b.	
c.	2.0 cc/g
d.	
e.	280°C
f.	
g.	
h.	

4. Heat of formation: $(\Delta H) \pm$ -188 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2390	cal/gm 2193	Aerojet Report No. 117A
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white, crystalline solid		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method) 1.64 (NOL)	gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color white	13. Odor none
14. pH at 25°C. 3.6	(Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) With Beckman meter, 0.024 M in acetone/water (5/1 volume fraction)		

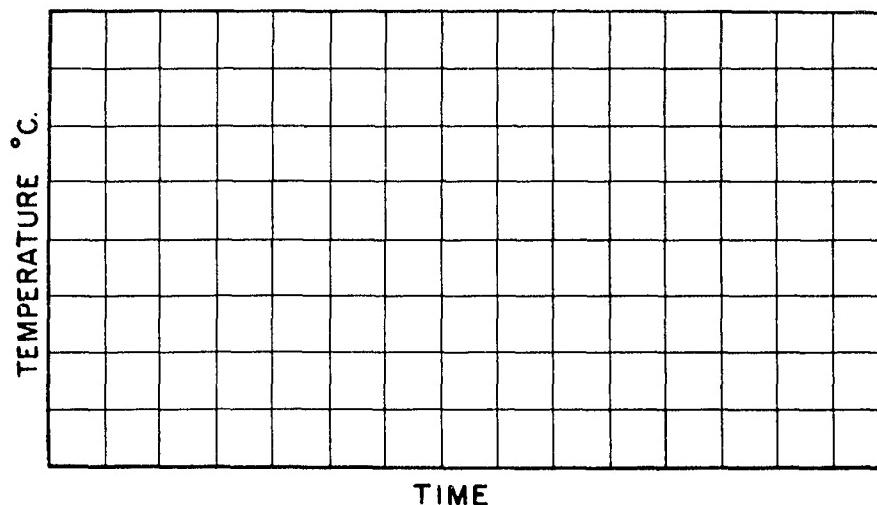


Ultraviolet Absorption Spectrum of 3,3-Dinitro 1,5-pentane
Diammonium Dinitrate in Methanol

20. Melting point: 151-153 °C.

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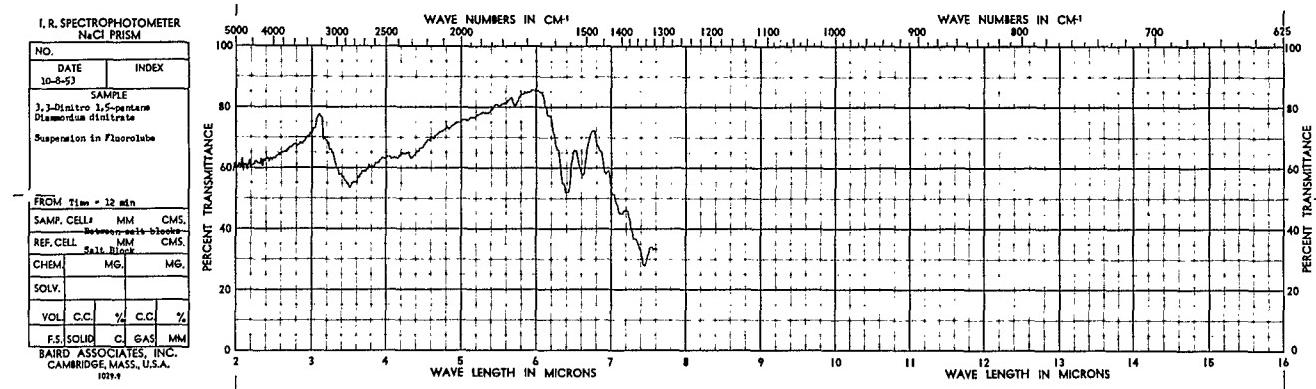
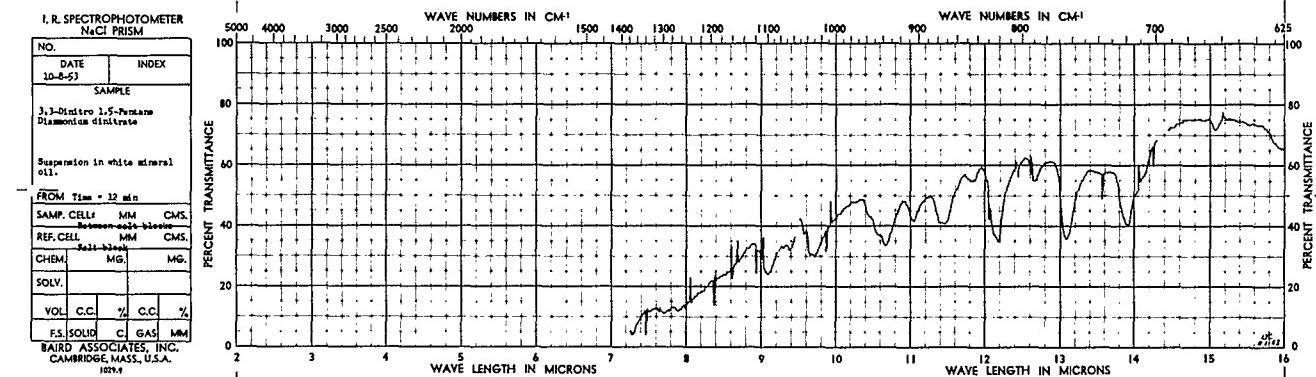
21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

260	g/100 ml H ₂ O at 25°C.	g/100 ml H ₂ O at	°C.
0.1	g/100 ml acetone	at	25 °C.
<0.1	g/100 ml toluene	at	25 °C.

(name material used as solvent)



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Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:

- (a) By itself _____
- (b) In mixtures (with additives) _____
- (c) Inhibiting action on polymerization of:

Thiokol _____

Methacrylate _____

Other compounds _____

30. Availability

- a. Amount now available? research quantities _____
- b. When was available material first prepared? _____
- c. Amount prepared at that time? _____
- d. Is large production feasible? _____
- e. Plant capacity in existence, lbs/day? _____
- f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 112

Calculated Ballistic-Mortar Value = 116

} Method of Aerojet Report No. 512, p.8.

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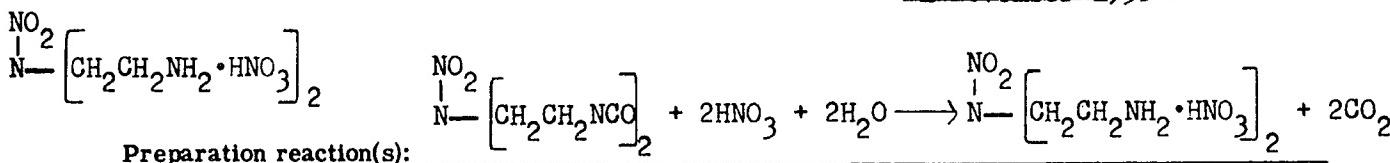
Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 3-Nitraza 1,5-pentane diammonium

Name dinitrate
Empirical formula C₂H₇N₆O₈
Structure: (configuration)

Information submitted by:
Activity Aerojet-General Corporation
Person M.R.Frankel and L.T.Carleton
Date 1 November 1953



1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	17.52	5.15		30.65		
By determination	17.66	5.11		30.78		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL X 5000 X 5000	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL X 5000 X 5000	48 hr at 100°C
d. Temperature of Explosion	OSRD 3401 p.6	
e. Temperature of Ignition	NOL X 5000 X 5000	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound
(designation-TNT, Tetryl, N.C., etc.)

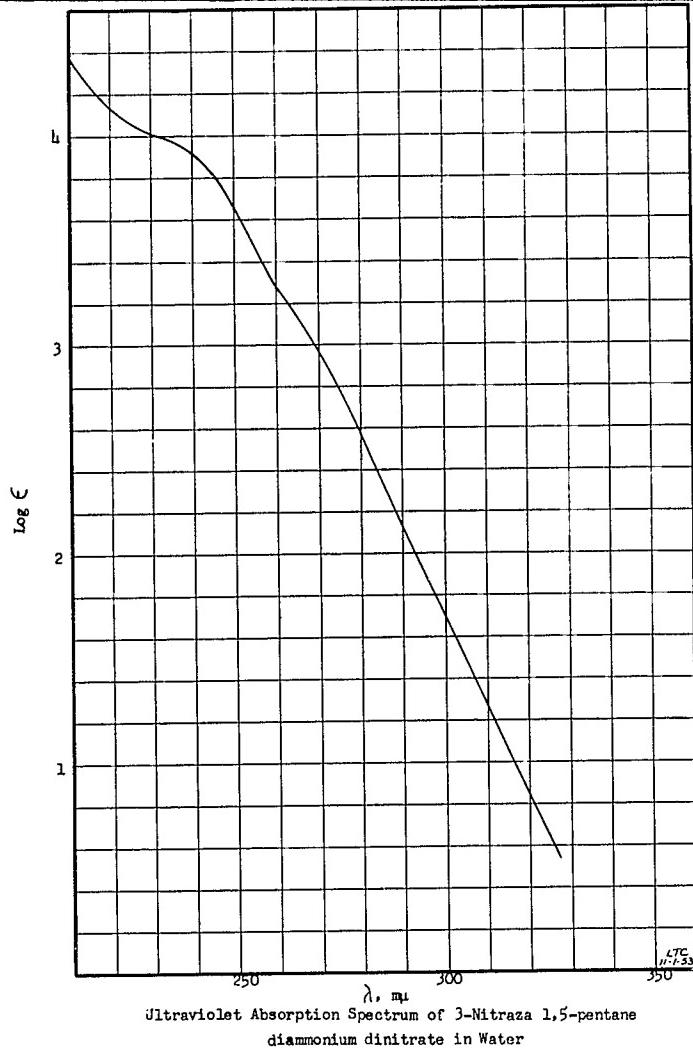
New Compound test results

a. <u>Tetryl, 32 cm/2.5 kg</u>	<u>61 cm/2.5 kg</u>
b.	
c.	<u>0.0 cc/g</u>
d.	
e.	<u>281°C</u>
f.	
g.	
h.	

4. Heat of formation: $(\Delta H) + -170$ Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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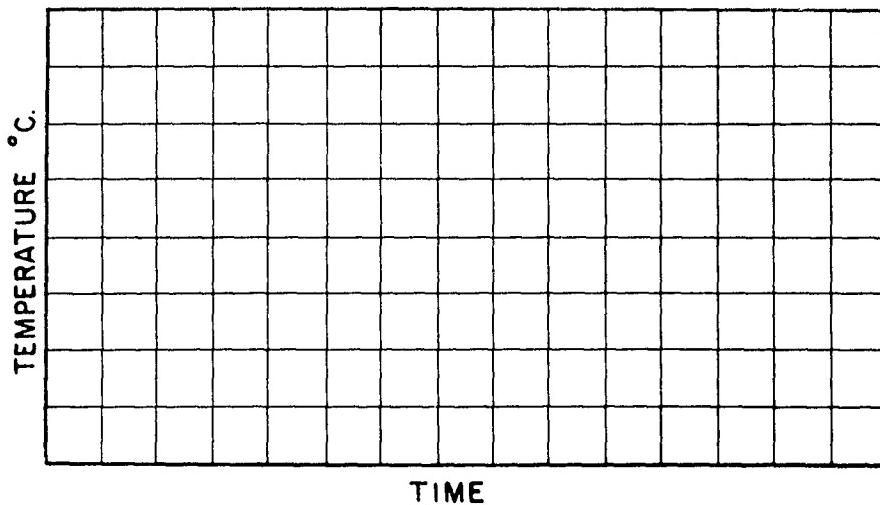
	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2496	cal/gm	2626
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	<u>white, crystalline solid</u>		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method)	1.57	gm/cm ³ . (Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
13. Odor	none		
14. pH at 25°C.	3.4	(Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)	
	With Beckman meter, saturated solution in acetone/water (5/1 volume ratio)		



20. Melting point: 153-155 °C.

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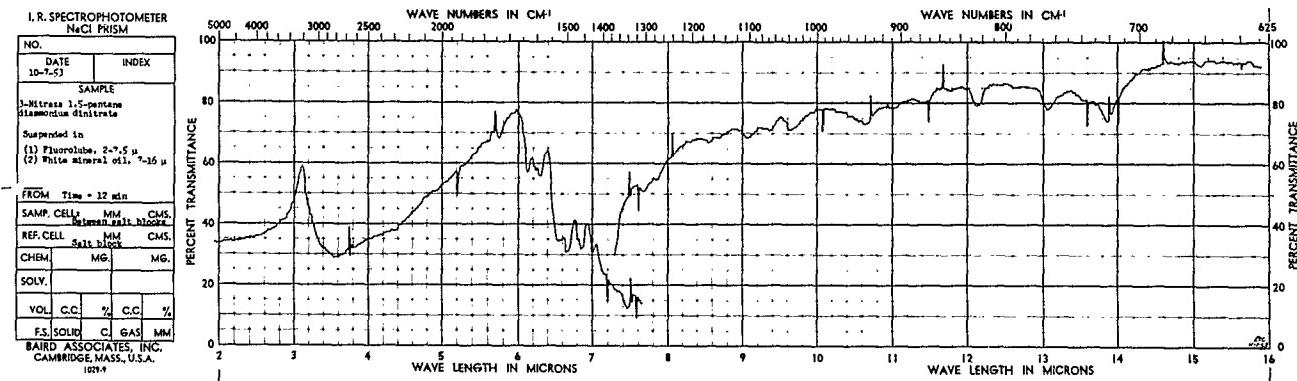
21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

120 g/100 ml H₂O at 25°C.
<0.1 g/100 ml acetone at 25°C.
<0.1 g/100 ml toluene at 25°C.

(name material used as solvent)



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Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
 (a) By itself _____
 (b) In mixtures (with additives) _____
 (c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____

30. Availability
 a. Amount now available? research quantities _____
 b. When was available material first prepared? _____
 c. Amount prepared at that time? _____
 d. Is large production feasible? _____
 e. Plant capacity in existence, lbs/day? _____
 f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 112)
 Calculated ballistic-mortar value = 126) Method of Aerojet Report No. 512, p.8.

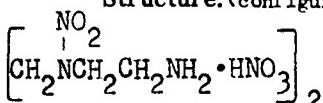
CONFIDENTIAL

Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

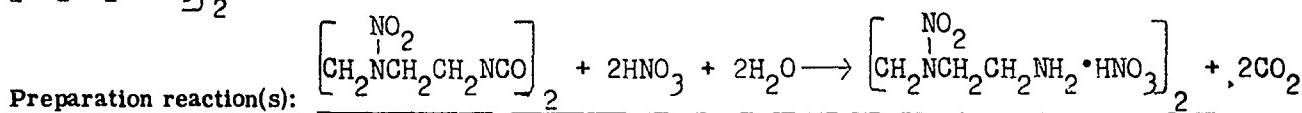
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COMPOUND: 3,6-Dinitraza 1,8-octane

Name diammonium dinitrate
Empirical formula C₆H₁₈N₈O₁₀
Structure: (configuration)



Information submitted by:
Activity Aerojet-General Corporation
Person M. B. Frankel and L.T. Carleton
Date 1 November 1953



Preparation reaction(s):

1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	19.89	5.01		30.93		
By determination	20.56	5.01		30.78		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method
a. Impact Sensitivity	NOL OSRD 3401 p.6
b. Thermal Stability	OSRD 3401 p.8
c. Vacuum Stability	NOL OSRD 3401 p.6
d. Temperature of Explosion	OSRD 3401 p.6
e. Temperature of Ignition	NOL OSRD 3401 p.6
f.	
g.	
h.	

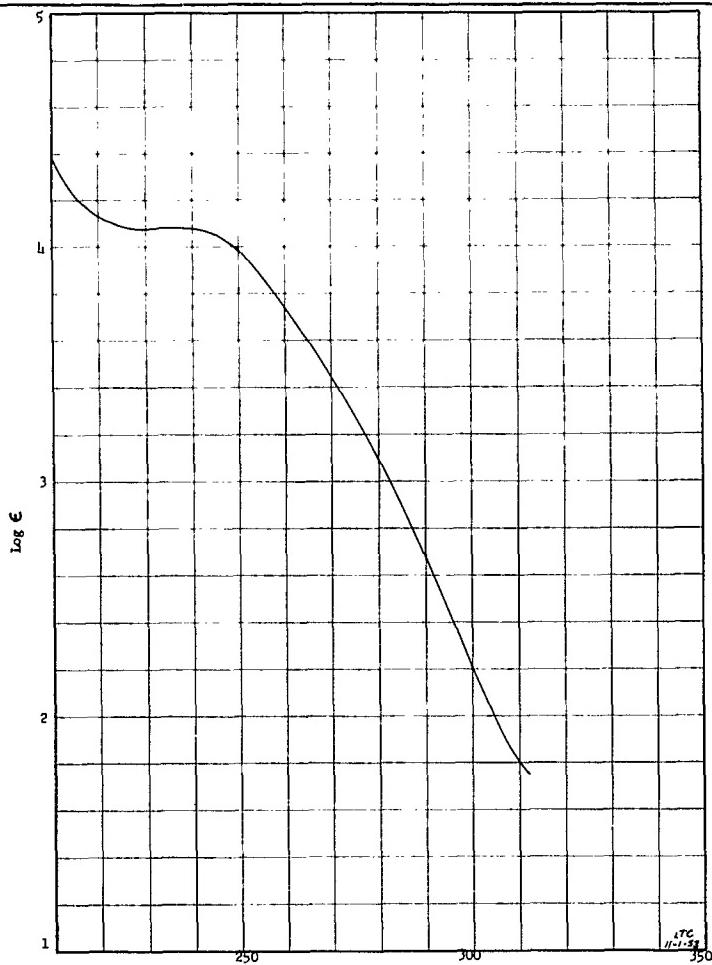
RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	58 cm/2.5 kg
b.	0.0 cc/g
c.	
d.	
e.	317°C
f.	
g.	
h.	

4. Heat of formation: (ΔH) + -159 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2816	cal/gm	2880
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)			white, crystalline solid
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method) 1.60 (NOL)	gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
14. pH at 25°C. 3.9	(Method reference OSRD 3401 o.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)	With Beckman meter.	
	saturated solution in acetone/water (5/1 volume ratio)		

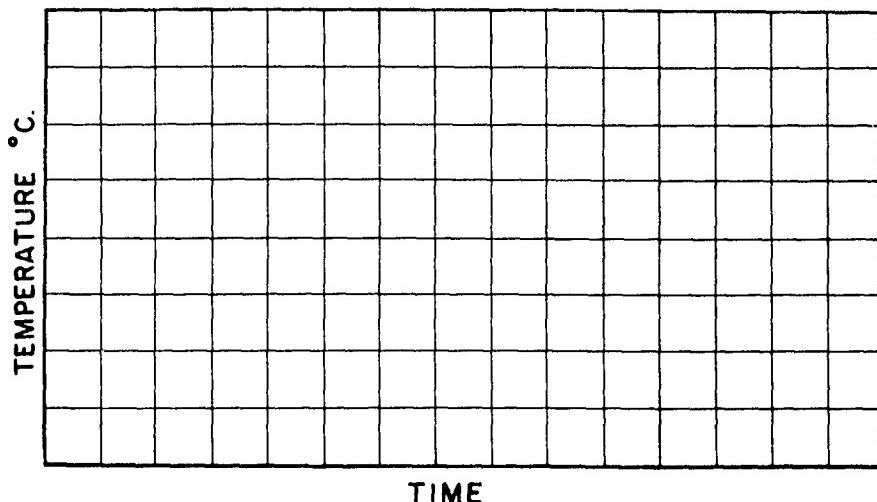


λ, μ
Ultraviolet Absorption Spectrum of 3,6-Dinitraza 1,8-Octane
Diammonium Dinitrate in Water

20. Melting point: 185-190 °C.

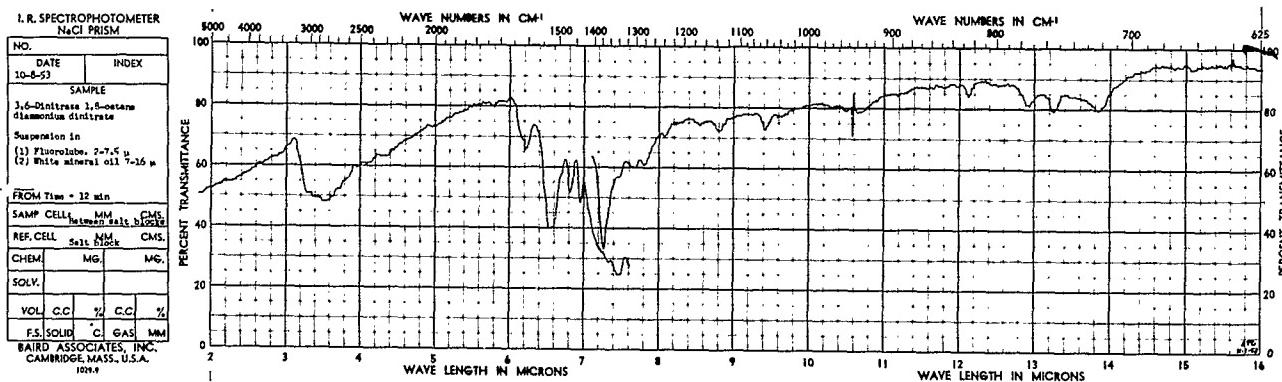
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21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

1.1 g/100 ml H₂O at 25°C. g/100 ml H₂O at _____ °C.
 <0.1 g/100 ml acetone at 25 °C.
 <0.1 g/100 ml toluene at 25 °C.
 (name material used as solvent)



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Report No. 770
SPIA/M3

Under compatability we are considering the ability of two compounds to be in intimate contact (Note CSDR 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thickol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? _____ research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existance, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____
- Calculated lead-block value = 109
Calculated ballistic-mortar value = 125 } Method of Aerojet Report No. 512. p.8.
- _____

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Data Questionnaire on

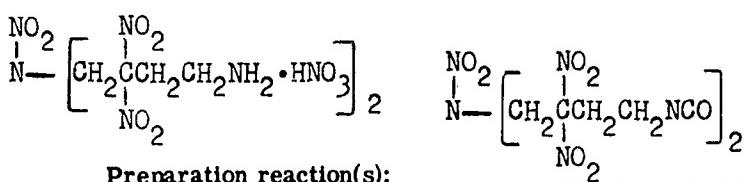
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, API/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

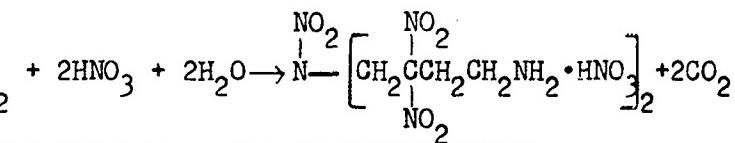
COMPOUND: 3,3,5,7,7-Pentanitro 5-aza

Name 1,9-Nonane diammonium dinitratoEmpirical formula C₈H₁₈N₁₀O₁₆

Structure: (configuration)



Information submitted by:

Activity Aerojet-General CorporationPerson M.B.Frankel and L.T.CarletonDate 1 November 1953

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen	
Calculated from formula	18.83	3.56		27.45	
By determination	18.95	3.88		27.10	

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method
a. Impact Sensitivity	NOL OSRD XIX
b. Thermal Stability	OSRD 3401 p.8
c. Vacuum Stability	NOL OSRD IXHEKPMX
d. Temperature of Explosion	OSRD 3401 p.6
e. Temperature of Ignition	NOL OSRD XIXKPK
f.	
g.	
h.	

2.5-kg weight

48 hr at 100°C

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, N.C., etc.)

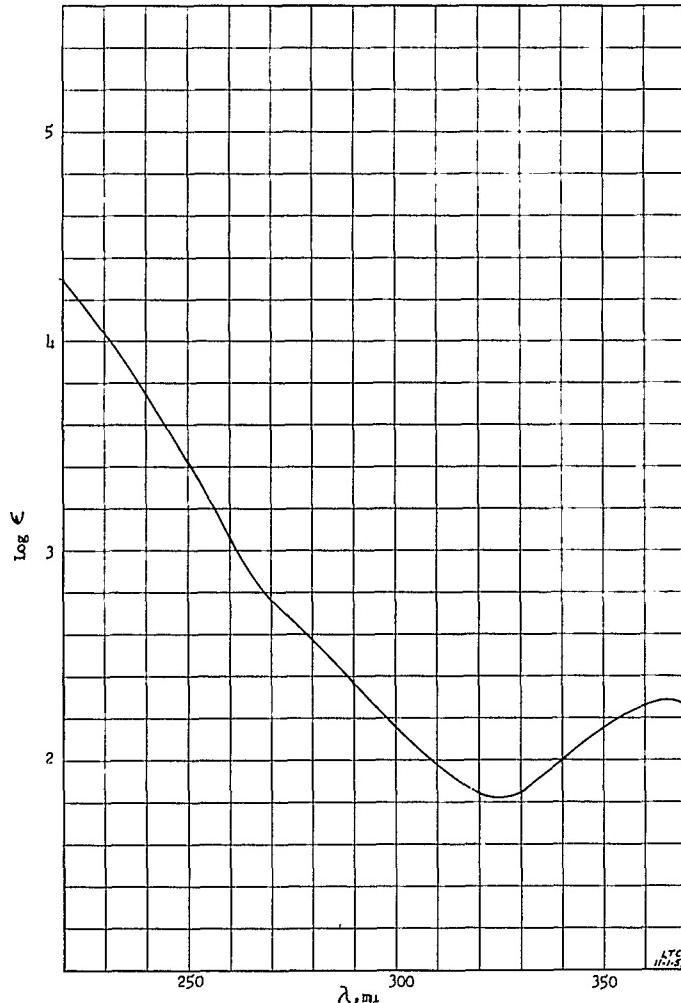
New Compound test results

2. Tetryl. 32 cm/2.5 kg	37 cm/2.5 kg
b.	
c.	Over 30 cc/g/48 hr
d.	
e.	197°C
f.	
g.	
h.	

4. Heat of formation: (ΔH) -209 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

CONFIDENTIAL

	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _f) (at 25°C. H ₂ O liquid)	2270	cal/gm	2353
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white needle-shaped crystals		
9. Simple microscope analysis data: (crystal studies)			
10. Density(Macro method) (NOL)	1.86 gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
14. pH at 25°C. solvent and concentrations used.	4.0*	pH indicator paper or Beckman pH meter.)	With Beckman meter, in saturated aqueous solution



Ultraviolet Absorption Spectrum of 3,3,5,7,7-Pantanitro-5-aza-1,9-Nonane Diammonium Dinitrate in Water

20. Melting point: 178-183 °C. (d)

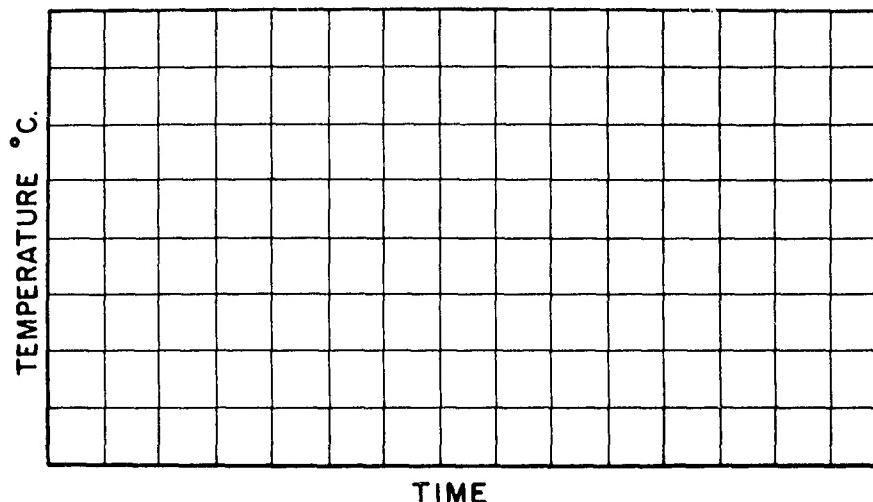
* Decreasing slowly.

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Appendix

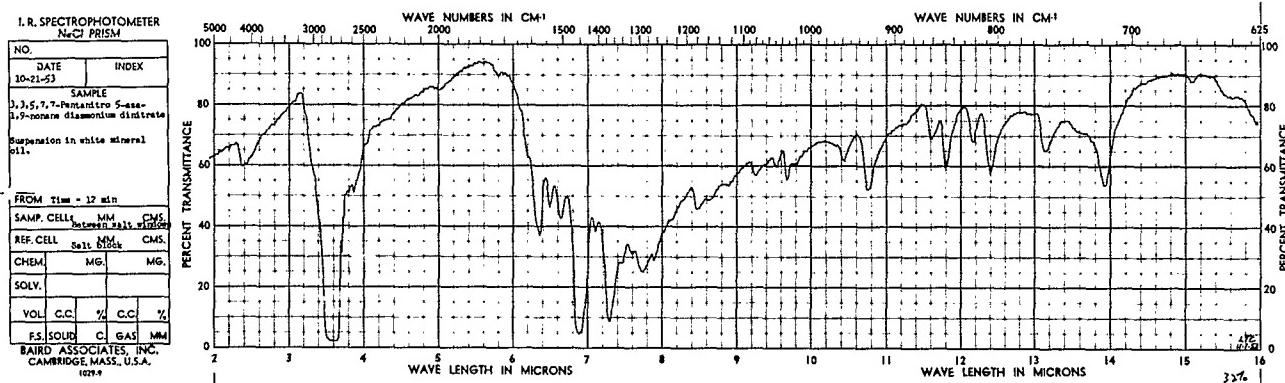
CONFIDENTIAL

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

0.2 g/100 ml H₂O at 25°C. g/100 ml H₂O at _____ °C.
 <0.1 g/100 ml acetone at 25 °C.
 <0.1 g/100 ml toluene at 25 °C.
 (name material used as solvent)



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Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____: _____

29. Polymerizing properties of the new compound:
 (a) By itself _____
 (b) In mixtures (with additives) _____
 (c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____
30. Availability
 a. Amount now available? _____ research quantities
 b. When was available material first prepared? _____
 c. Amount prepared at that time? _____
 d. Is large production feasible? _____
 e. Plant capacity in existance, lbs/day? _____
 f. Outline steps for a quantity production method

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____
- Calculated lead-block value = 135 Method of Aerojet Report No. 512, p.8.
 Calculated ballistic-mortar value = 11.1
- _____

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Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

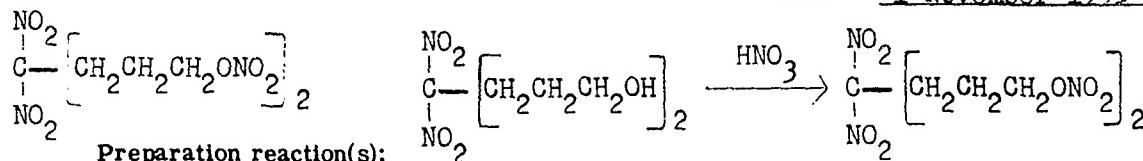
Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound further. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,7-Dinitrato 4,4-dinitro

Name heptaneEmpirical formula C₇H₁₂N₂O₁₀

Structure: (configuration)

Information submitted by:

Activity Aerojet-General CorporationPerson M.B.Frankel and L.T.CarletonDate 1 November 1953

Preparation reaction(s):

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	26.93	3.88		17.95		
By determination	27.17	4.15		18.40		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method
a. Impact Sensitivity	OSRD 3185
b. Thermal Stability	OSRD 3401 p.8
c. Vacuum Stability	OSRD 3401 p.10
d. Temperature of Explosion	OSRD 3401 p.6
e. Temperature of Ignition	OSRD 3401 p.6
f.	
g.	
h.	

RESULTS OF ABOVE TESTS

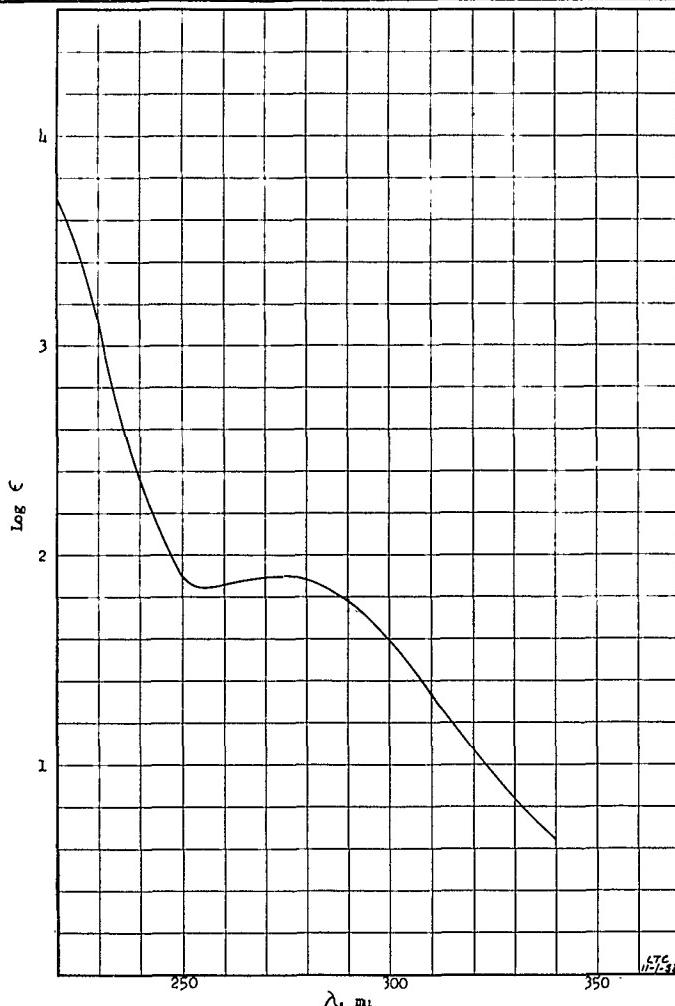
Reference compound (designation-TNT, Tetryl, N.C., etc.) New Compound test results

a.	
b.	
c.	
d.	
e.	
f.	
g.	
h.	

4. Heat of formation: (ΔH) + -127 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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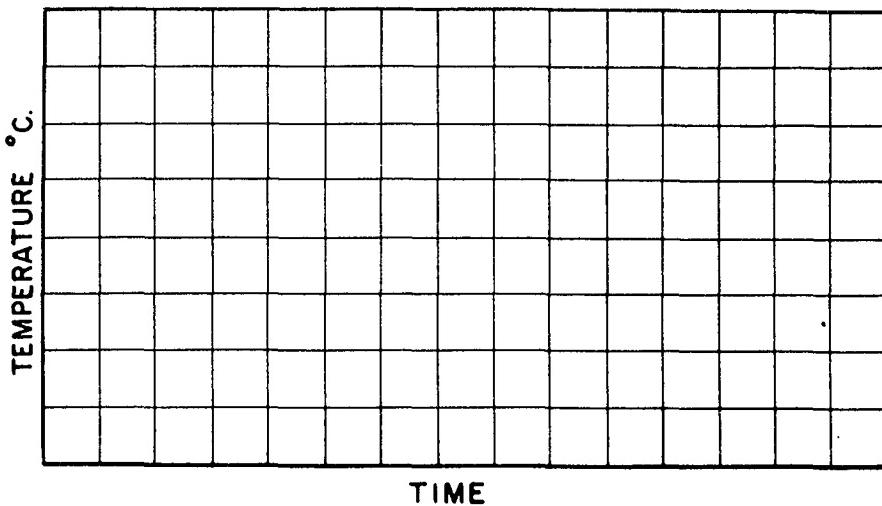
	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	3015	cal/gm	3103
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	<u>flaky white crystals</u>		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method)	gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction (n _D ^{25°C.})		12. Color	white
14. pH at 25°C. 8.6	(Method reference OSRD 3401 o.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used, pH indicator paper or Beckman pH meter.) With Beckman meter, 0.008 M in acetone/water (5/1 volume ratio)		



Ultraviolet Absorption Spectrum of 1,7-Dinitro-1,1-dinitroheptane in Methanol

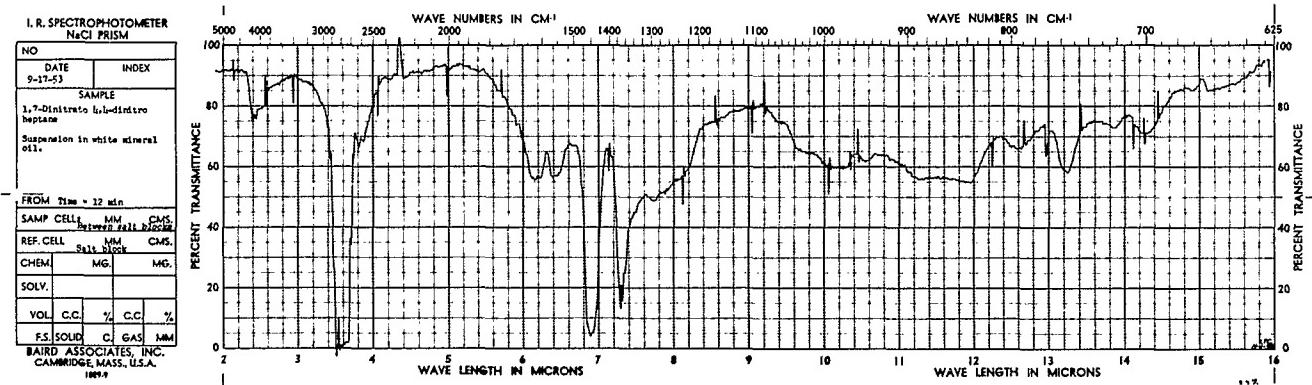
20. Melting point 28 °C.

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



- ## **22. Solubility of new compound:**

<0.1 g/100 ml H₂O at 25°C. g/100 ml H₂O at °C.
nearly ∞ g/100 ml acetone at 25 °C.
(name material used as solvent)
nearly ∞ g/100 ml toluene at 25 °C.
(name material used as solvent)



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SPLA/M3

Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:

(a) By itself _____

(b) In mixtures (with additives) _____

(c) Inhibiting action on polymerization of:

Thiokol _____

Methacrylate _____

Other compounds _____

30. Availability

a. Amount now available? research quantities

b. When was available material first prepared?

c. Amount prepared at that time?

d. Is large production feasible?

e. Plant capacity in existence, lbs./day?

f. Outline steps for a quantity production method

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 120 Method of Aerojet Report No. 512. p.8.
 Calculated ballistic-mortar value = 132

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Date Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

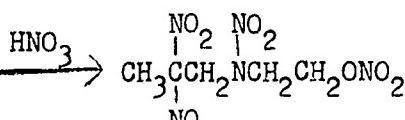
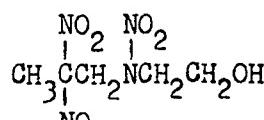
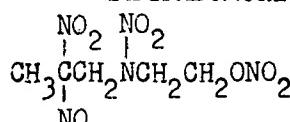
Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1-Nitrato-3,5,5-trinitro-3-aza-

Name hexane

Empirical formula C₅H₉N₅O₉

Structure:(configuration)



Information submitted by:

Activity Aerojet-General Corporation

Person M.B.Frankel and L.T.Carleton

Date 1 November 1953

Preparation reaction(s):

1. Quantitative analysis:(% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	21.21	3.20		24.74		
By determination	21.74	3.51		25.31		

2. Burning properties:(compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL	2.5-kg weight
b. Thermal Stability		
c. Vacuum Stability	NOL	100°C
d. Temperature of Explosion		
e. Temperature of Ignition	OSRD 3401 p.6	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, N.C., etc.)

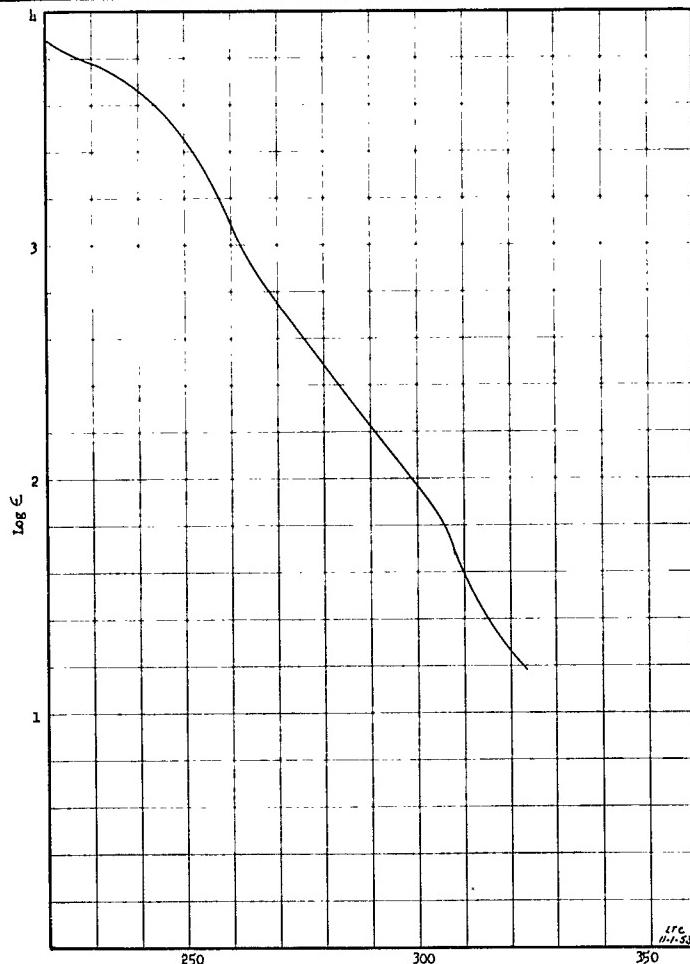
New Compound test results

a. Tetryl, 32 cm/2.5 kg	21 cm/2.5 kg
b.	
c.	1.1 cc/g/48 hr; 1.5 cc/g/120 hr
d.	
e.	
f.	
g.	
h.	

4. Heat of formation: (ΔH_f) + -73 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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	By Experiment	By Calculation	Method
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	Description or reference. Separate sheet if necessary.
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2487	cal/gm	2533
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white, crystalline solid		
9. Simple microscope analysis data: (crystal studies)			
10. Density(Macro method) (NOL)	1.73 gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
14. pH at 25°C.	(Method reference OSRD 3401 o.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)	With Beckman meter, 0.019 M in acetone/water (5/1 volume ratio)	

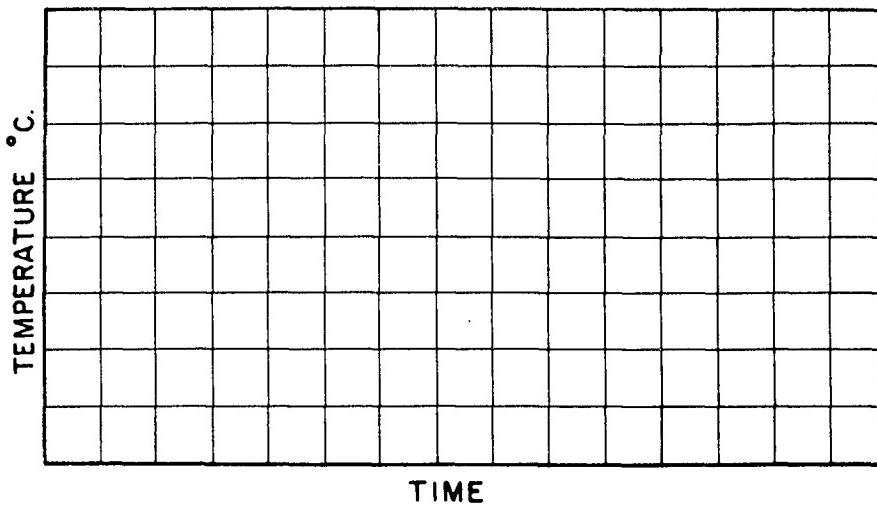


Ultraviolet Absorption Spectrum of 1-Nitroso-3,5,5-trinitro
3-aza-Hexane in Methanol

20. Melting point: 116-117 °C.

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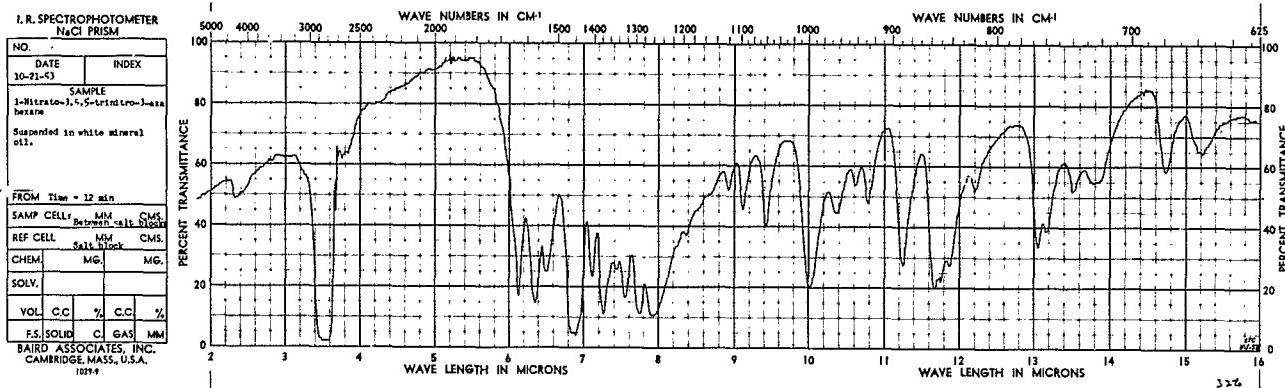
21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H ₂ O at 25°C.	g/100 ml H ₂ O at _____ °C.
23	g/100 ml acetone	at 25 °C.
0.2	g/100 ml toluene	at 25 °C.

(name material used as solvent)



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Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
 (a) By itself _____
 (b) In mixtures (with additives) _____
 (c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____
30. Availability
 a. Amount now available? _____ research quantities
 b. When was available material first prepared? _____
 c. Amount prepared at that time? _____
 d. Is large production feasible? _____
 e. Plant capacity in existence, lbs./day? _____
 f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____
- Calculated lead-block value = 1142 } Method of Aerojet Report No. 512, p.8
 Calculated ballistic-mortar value = 1146 }
- _____

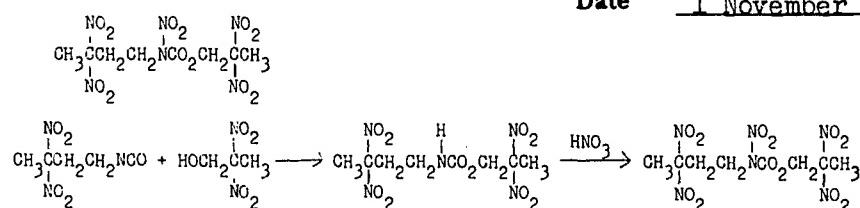
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Date Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 2,2,6,9,9-Pantanitro-4-oxa-5-keto

Name 6-aza-decane
Empirical formula C₈H₁₂N₆O₁₂
Structure:(configuration)



Information submitted by:
Activity Aerojet-General Corporation
Person M.B.Frankel and L.T.Carleton
Date 1 November 1953

1. Quantitative analysis(% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	25.01	3.15		21.88		
By determination	25.96	3.36		21.73		

2. Burning properties:(compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used.
(Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD X18X	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL OSRD X18X(p.10)	100°C
d. Temperature of Explosion	OSRD 3401 p.6	
e. Temperature of Ignition	NOL OSRD X18X(p.8X)	
f.		
g.		
h.		

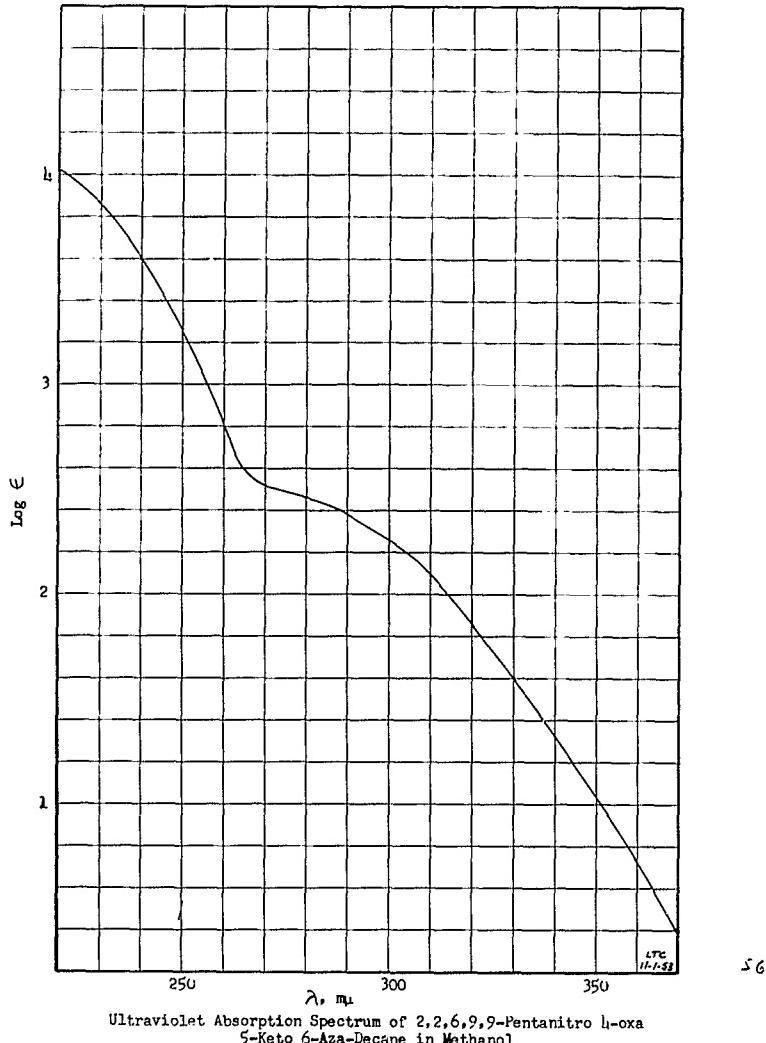
RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	47 cm/2.5 kg
b.	
c.	Class II
d.	
e.	293°C
f.	
g.	
h.	

4. Heat of formation: (ΔH) -147 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

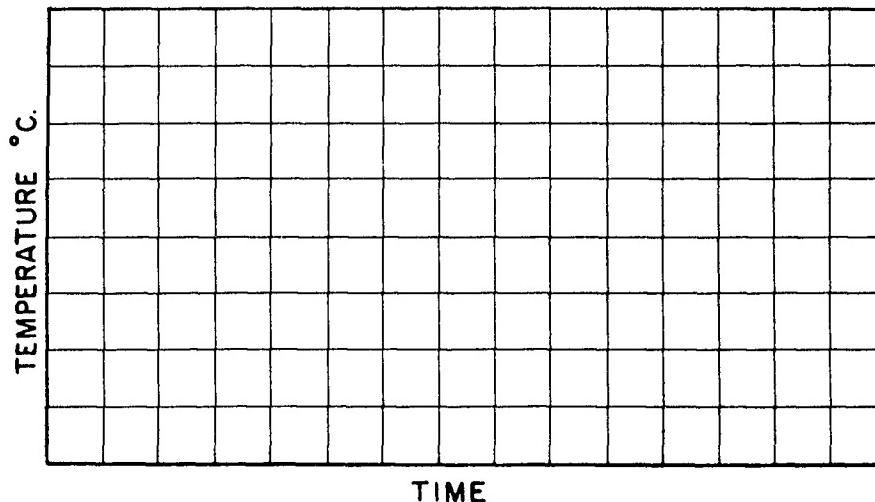
CONFIDENTIAL

	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2643	cal/gm	2666
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white crystalline solid		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method) 1.61 gm/cm ³ . (NOL)	gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
13. Odor	none		
14. pH at 25°C. solvent and concentrations used. pH indicator paper or Beckman pH meter.)	(Method reference OSRD 3401 o.4, or OSRD 5968. Indicate method used, i.e. 0.024 M in acetone/water (5/1 volume ratio)		With Beckman meter,



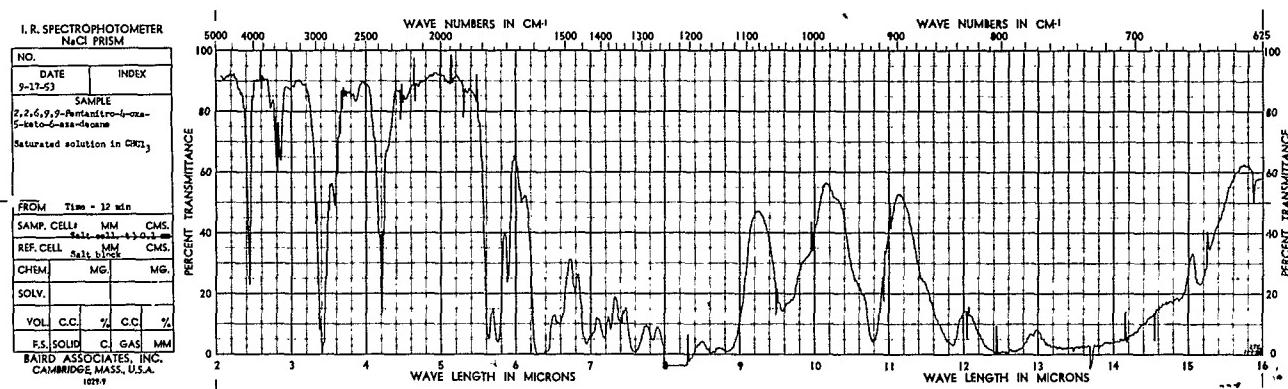
20. Melting point: 70-72 °C.

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H ₂ O at 25°C.	g/100 ml H ₂ O at	°C.
190	g/100 ml	acetone	at 25 °C.
4.3	g/100 ml	(name material used as solvent)	
		toluene	at 25 °C.
		(name material used as solvent)	



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Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? _____ research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existance, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____
- Calculated lead-block value = 117 Method of Aerojet Report No. 512, p. 8
Calculated ballistic-mortar value = 126

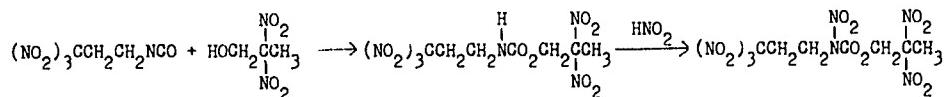
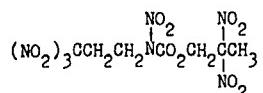
CONFIDENTIAL

Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 2,2,6,9,9,9-Hexanitro 4-oxa 5-keto

Name 6-aza-nonane
Empirical formula C₇H₉N₇O₇
Structure: (configuration)



Information submitted by:
Activity Aerojet-General Corporation
Person M.B.Frankel and L.T.Carleton
Date 1 November 1953

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	20.25	2.19		23.62		
By determination	20.51	2.22		21.05		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method
a. Impact Sensitivity	NOL OSRD 3401 p.5
b. Thermal Stability	OSRD 3401 p.8
c. Vacuum Stability	NOL OSRD 3401 p.10
d. Temperature of Explosion	OSRD 3401 p.6
e. Temperature of Ignition	NOL OSRD 3401 p.8
f.	
g.	
h.	

2.5-kg weight18 hr at 100°C

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, N.C., etc.)

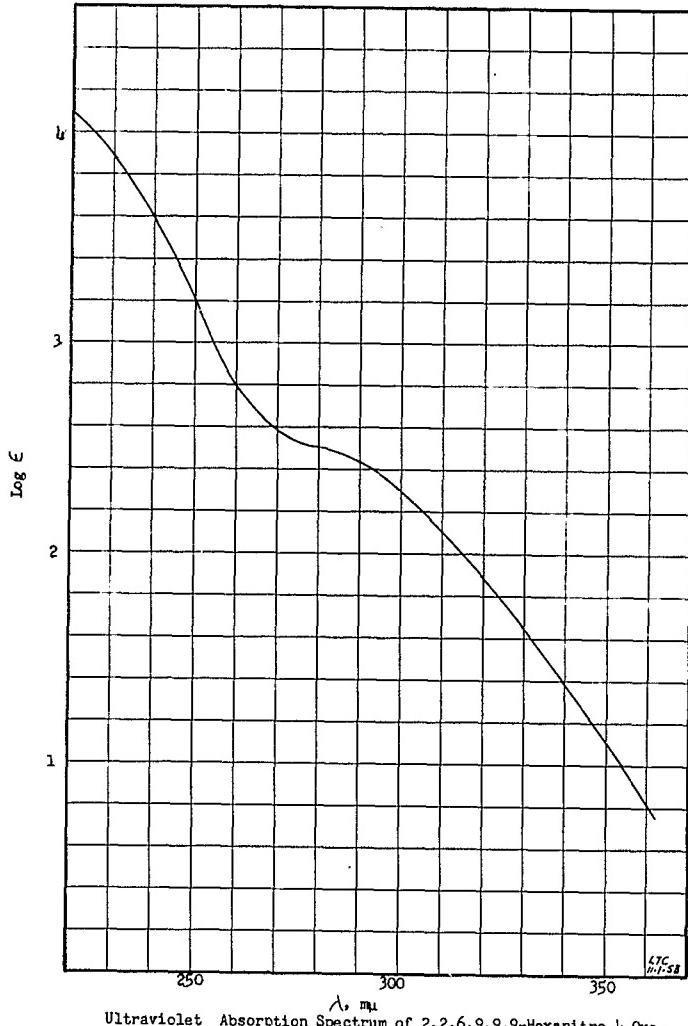
New Compound test results

a. Tetryl, 32 cm/2.5 kg	16 cm/2.5 kg
b.	
c.	Class III
d.	
e.	22°C
f.	
g.	
h.	

4. Heat of formation: (ΔH) + -114 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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	By Experiment	By Calculation	Method
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	Description or reference. Separate sheet if necessary.
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2051	cal/gm	2033 .
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white, needle-shaped crystals		
9. Simple microscope analysis data: (crystal studies)			
10. Density(Macro method)	gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
14. pH at 25°C.	4.5	(Method reference OSRD 3401 b.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)	With Beckman pH meter, 0.01M in acetone/water (5/1 volume ratio)

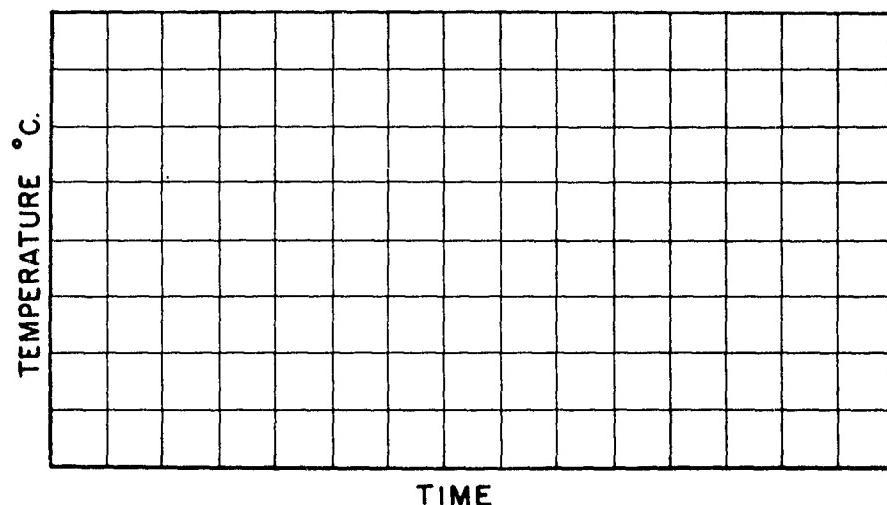


Ultraviolet Absorption Spectrum of 2,2,6,9,9,9-Hexanitro 4-Oxa-5-Keto-6-Aza-Nonane in Methanol

20. Melting point: 67 °C.

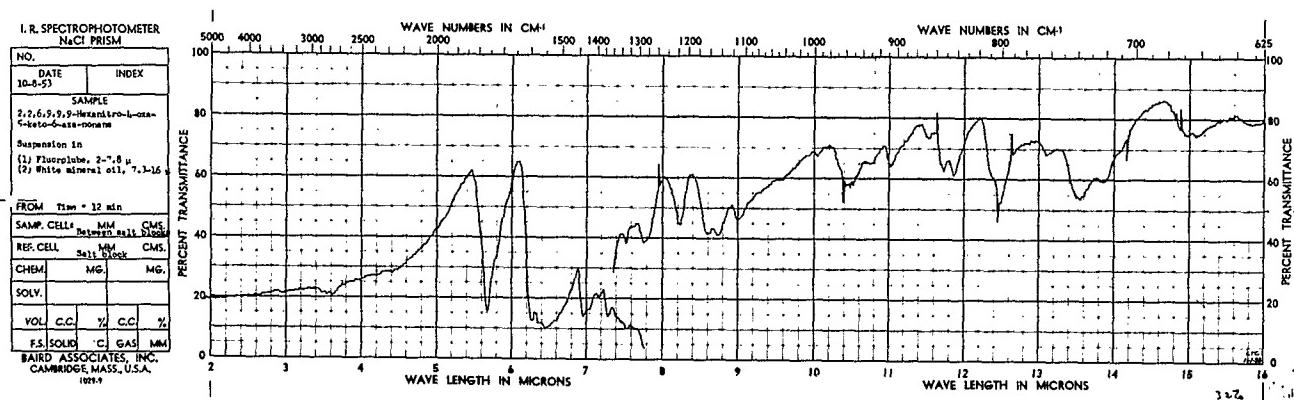
CONFIDENTIAL

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1 g/100 ml H₂O at 25°C. g/100 ml H₂O at °C.
212 g/100 ml acetone at 25 °C.
3.0 g/100 ml (name material used as solvent)
 toluene at 25 °C.
 (name material used as solvent)



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Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

_____26. Compatability with nitrocellulose: _____

_____27. Compatability with rubber: _____

_____28. Compatability with _____: _____

29. Polymerizing properties of the new compound:

- (a) By itself _____
- (b) In mixtures (with additives) _____
- (c) Inhibiting action on polymerization of:
Thiokol _____
Methacrylate _____
Other compounds _____

30. Availability

- a. Amount now available? research quantities
 - b. When was available material first prepared?
 - c. Amount prepared at that time?
 - d. Is large production feasible?
 - e. Plant capacity in existence, lbs/day?
 - f. Outline steps for a quantity production method
-
- _____
-
- _____
-
- _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 153

Method of Aerojet Report No. 512, p.8.

Calculated ballistic mortar value = 110

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Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

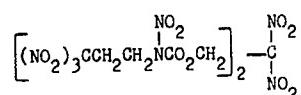
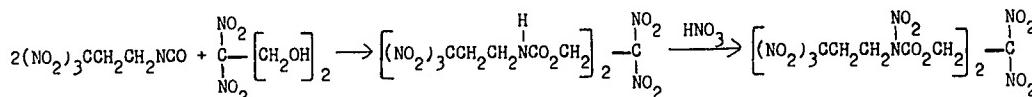
COMPOUND: 1,1,1,4,8,8,12,15,15,15-Decanitro 4,12-diaza

Name 5,11-diketo-6,10-dioxa-pentadecane

Information submitted by:

Empirical formula C₁₁H₁₂N₂O₂Activity Aerojet-General Corporation

Structure: (configuration)

Person M.B. Frankel and L.T. CarletonDate 1 November 1953

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	18.97	1.71		24.11		
By determination	19.29	2.00		24.21		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet	Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)
Name of test	Recommended method
a. Impact Sensitivity	NOL OSRD 3401 p.5X
b. Thermal Stability	OSRD 3401 p.8
c. Vacuum Stability	NOL OSRD 3401 p.10
d. Temperature of Explosion	OSRD 3401 p.6
e. Temperature of Ignition	NOL OSRD 3401 p.15X
f.	
g.	
h.	

RESULTS OF ABOVE TESTS

Reference compound _____ New Compound test results

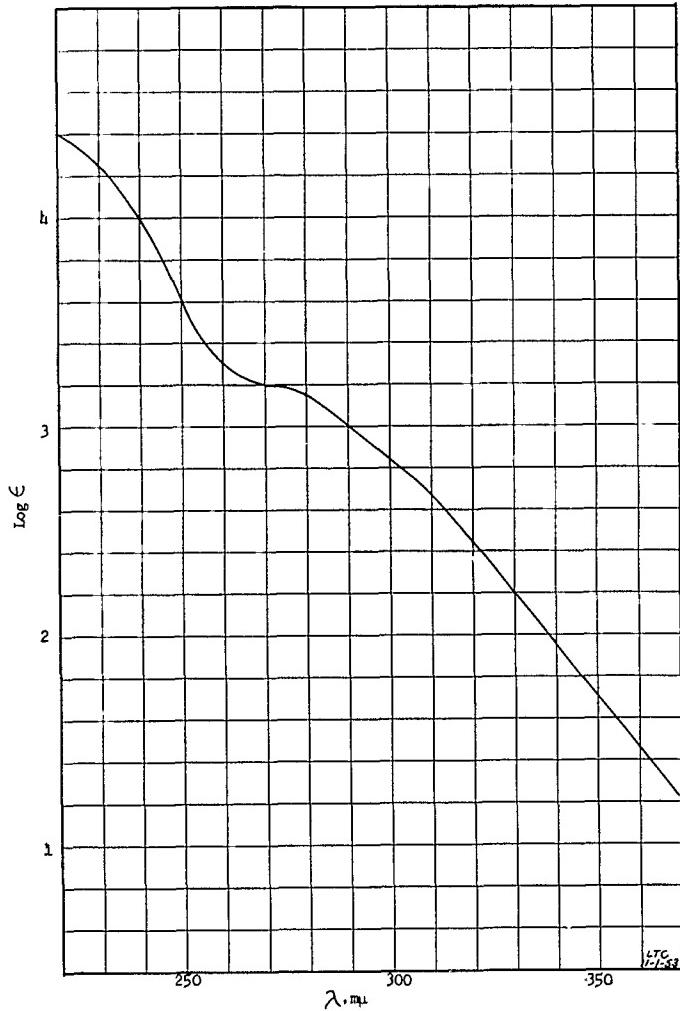
(designation-TNT, Tetryl, N.C., etc.)

a. Tetryl, 32 cm/2.5 kg	20 cm/2.5 kg
b.	5.0 cc/g
c.	
d.	
e.	212°C
f.	
g.	

4. Heat of formation: (ΔH) -167 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	1834	cal/gm	1784
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white, crystalline solid		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method) 1.74 gm/cm ³ . (NOL) (Micro or other method)		gm/cm ³ .	(Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	White
14. pH at 25°C. 5.9*		13. Odor	none
	(Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)		With Beckman meter, 0.009 M in acetone/water (5/1 volume ratio)



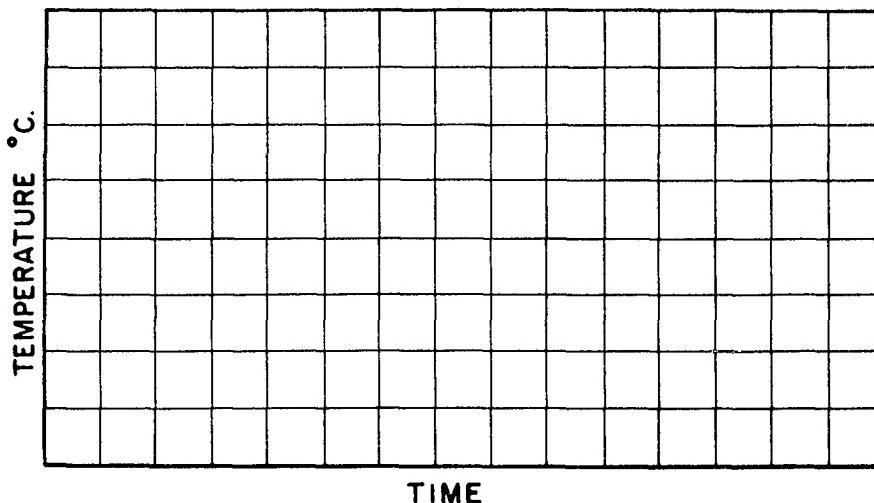
Ultraviolet Absorption Spectrum of 1,1,1,4,8,8,12,15,15,15-Decanitro
4,12-diaza 5,11-diketo 6,10-dioxa-pentadecane in Methanol

20. Melting point: 94-96 °C.

* Decreasing rapidly.

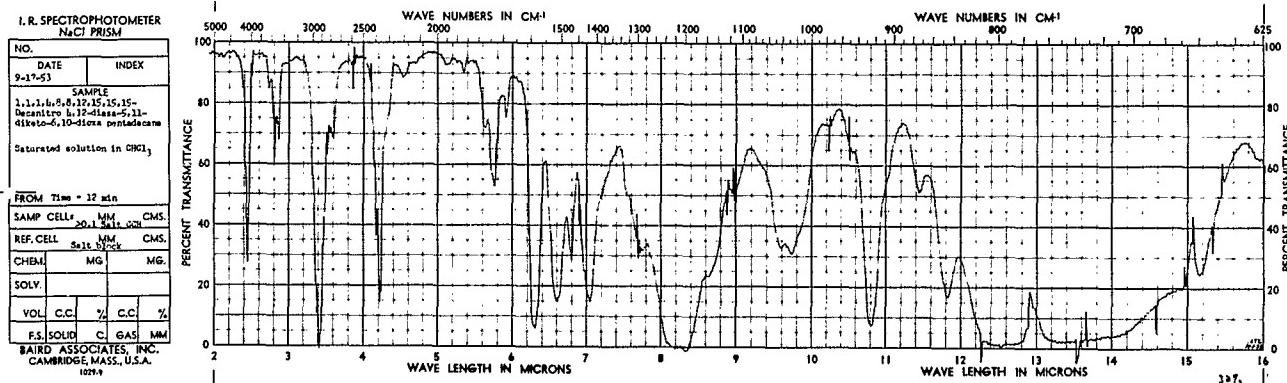
CONFIDENTIAL

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound.

<0.1 g/100 ml H₂O at 25°C. g/100 ml H₂O at _____ °C.
250 g/100 ml acetone at 25 °C.
g/100 ml _____ (name material used as solvent) at _____ °C.



CONFIDENTIAL

Report No. 770
SPLA/M3

Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:

- (a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
Thiokol _____
Methacrylate _____
Other compounds _____

30. Availability

- a. Amount now available? research quantities
b. When was available material first prepared?
c. Amount prepared at that time?
d. Is large production feasible?
e. Plant capacity in existence, lbs/day?
f. Outline steps for a quantity production method

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 164

Calculated ballistic-mortar value = 132

{ Method of Aerojet Report No. 512. p.8

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Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, API/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,1,1,5,8,11,15,15,15-Nonanitro 3,13-dioxa

Name 1,12-diketo 5,8,11-triaza-pentadecane Information submitted by:

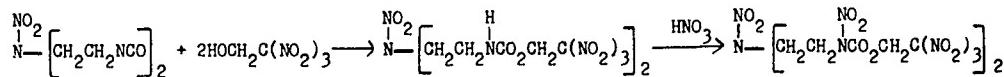
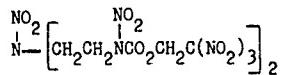
Empirical formula C₁₀H₁₂N₂O₂

Structure: (configuration)

Activity Aerojet-General Corporation

Person M.B.Frankel and L.T.Carleton

Date 1 November 1953



1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	<u>18.41</u>	<u>1.86</u>	<u>25.77</u>	<u>25.87</u>		
By determination	<u>18.98</u>	<u>2.23</u>				

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD 3401 p.8	<u>2.5-kg weight</u>
b. Thermal Stability	OSRD 3401 p.8	<u>48 hr at 100°C</u>
c. Vacuum Stability	NOL OSRD 3401 p.8	
d. Temperature of Explosion	OSRD 3401 p.8	
e. Temperature of Ignition	NOL OSRD 3401 p.8	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.) New Compound test results

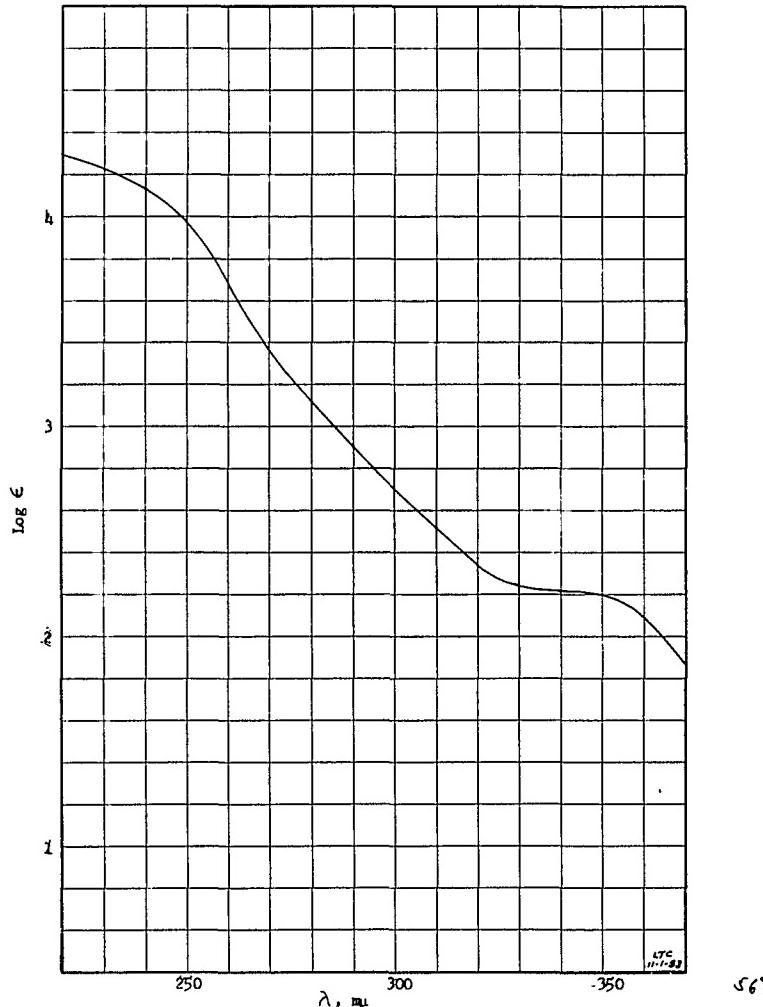
a. <u>Tetryl, 32 cm/2.5 kg</u>	<u>17 cm/2.5 kg</u>
b.	
c.	<u>1.1 cc/g</u>
d.	
e.	<u>220°C</u>
f.	
g.	
h.	

4. Heat of formation: (ΔH) -159 Kg. calories at 25°C., 1 atm. pressure

(Indicate sign)

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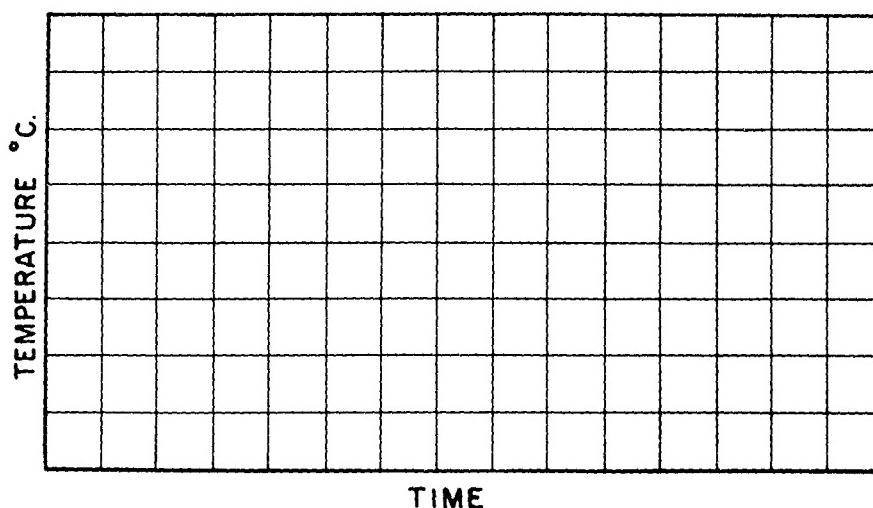
	By Experiment	By Calculation	Method
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	Description or reference. Separate sheet if necessary.
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	1826	cal/gm	1792
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	<u>white, crystalline solid</u>		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method)	1.77 gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
13. Odor	slight		
14. pH at 25°C. (Method reference OSRD 3401 o.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)	With Beckman meter, 0.008 M in acetone/water (5/1 volume ratio)		



Ultraviolet Absorption Spectrum of 1,1,1,5,8,11,15,15,15-Nonenitro
3,13-dioxa 4,12-diketo 5,8,11-triaza-pentadecane in Methanol

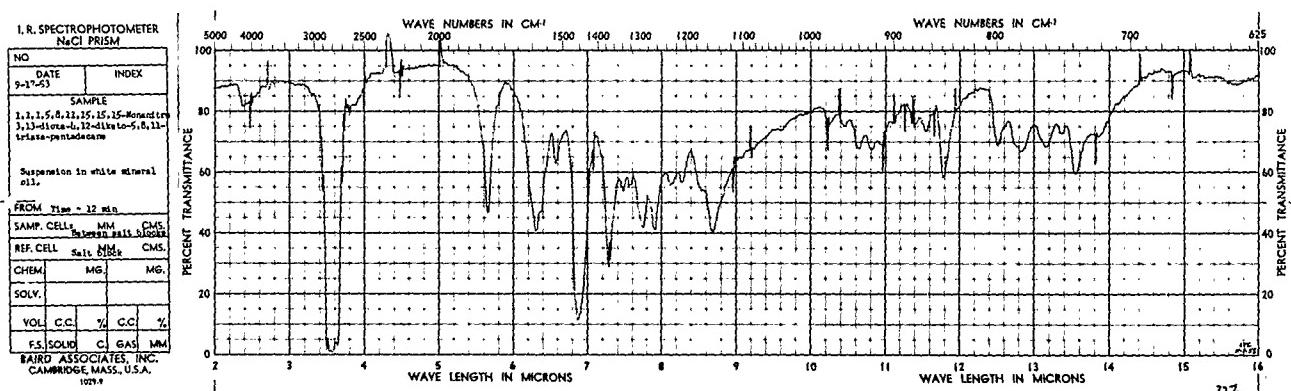
20. Melting point: 120-122 °C.

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1 g/100 ml H₂O at 25°C. g/100 ml H₂O at _____ °C.
100 g/100 ml acetone at 25 °C.
(name material used as solvent)
<0.1 g/100 ml toluene at 25 °C.
(name material used as solvent)



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Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____ :

29. Polymerizing properties of the new compound:

(a) By itself _____

(b) In mixtures (with additives) _____

(c) Inhibiting action on polymerization of:

Thiokol _____

Methacrylate _____

Other compounds _____

30. Availability

a. Amount now available? research quantities

b. When was available material first prepared? _____

c. Amount prepared at that time? _____

d. Is large production feasible? _____

e. Plant capacity in existence, lbs/day? _____

f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 162

Calculated ballistic-mortar value = 140

Method of Aerojet Report No. 512, p.8

CONFIDENTIAL

Date Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

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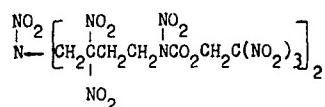
1,1,1,5,8,8,10,12,12,15,19,19,19-

COMPOUND: Tridecanitro 3,17-dioxa-4,16-diketo

Name 5,10,15-triaza-nonadecane

Empirical formula C₁₁H₁₆N₂O₃₀

Structure:(configuration)

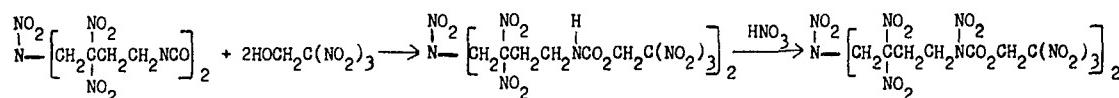


Information submitted by:

Activity Aerojet-General Corporation

Person M.B.Frankel and L.T.Carleton

Date 1 November 1953



1. Quantitative analysis(% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	<u>18.93</u>	<u>1.82</u>		<u>25.23</u>		
By determination	<u>19.12</u>	<u>1.84</u>		<u>25.19</u>		

2. Burning properties:(compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL <u>OSRD 3403</u>	<u>2.5-kg weight</u>
b. Thermal Stability		<u>OSRD 3401 p.8</u>
c. Vacuum Stability	NOL <u>OSRD 3404 EK BXW</u>	<u>18 hr at 100°C</u>
d. Temperature of Explosion		<u>OSRD 3401 p.6</u>
e. Temperature of Ignition	NOL <u>OSRD 3401 p.8</u>	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound
(designation-TNT, Tetryl, N.C., etc.)

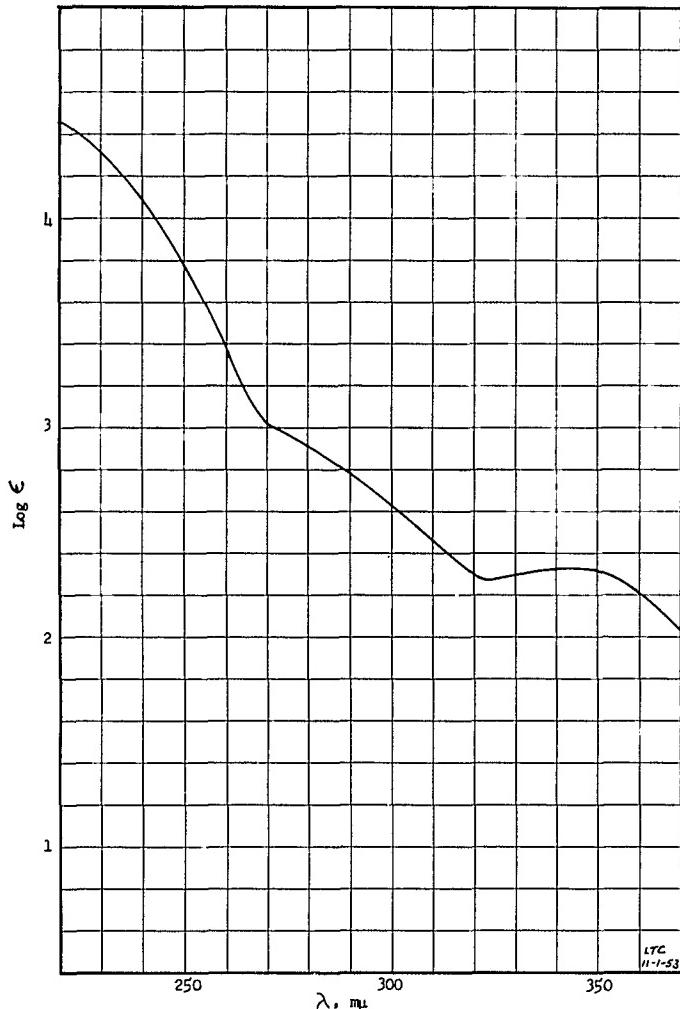
New Compound test results

a. <u>Tetryl, 32 cm/2.5 kg</u>	<u>12 cm/2.5 kg</u>
b.	
c.	<u>1.4 cc/g</u>
d.	
e.	<u>217°C</u>
f.	
g.	
h.	

4. Heat of formation: (ΔH) + -130 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

CONFIDENTIAL

	By Experiment	By Calculation	Method
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	Description or reference. Separate sheet if necessary.
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	1951	cal/gm	1859
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)			
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method) (NOL)	1.76 gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	13. Odor
14. pH at 25°C. 2.9	(Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)	In Beckman pH meter, 0.006 M in acetone/water (5/1 volume ratio)	

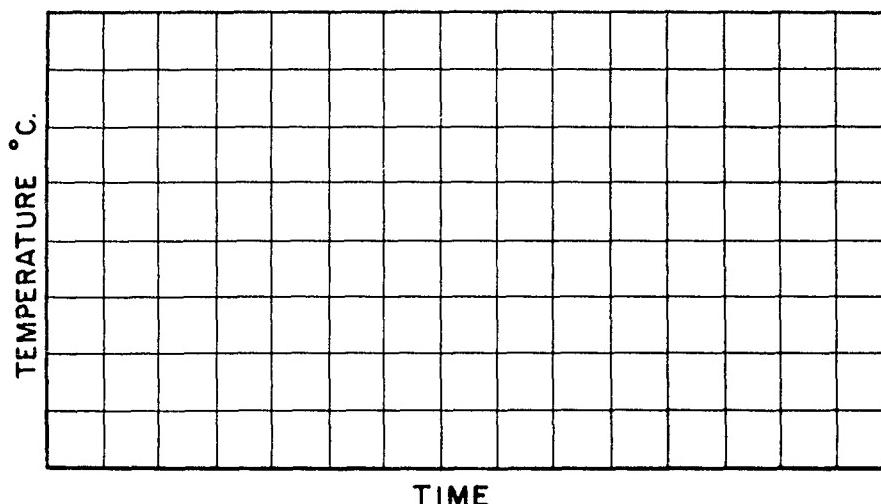


Ultraviolet Absorption Spectrum of 1,1,1,5,8,8,10,12,12,15,19,19,19-
Tridecanitro 3,17-dioxa 4,16-diketo 5,10,15-triaza-nonadecane in Methanol

20. Melting point: 150-151 °C.

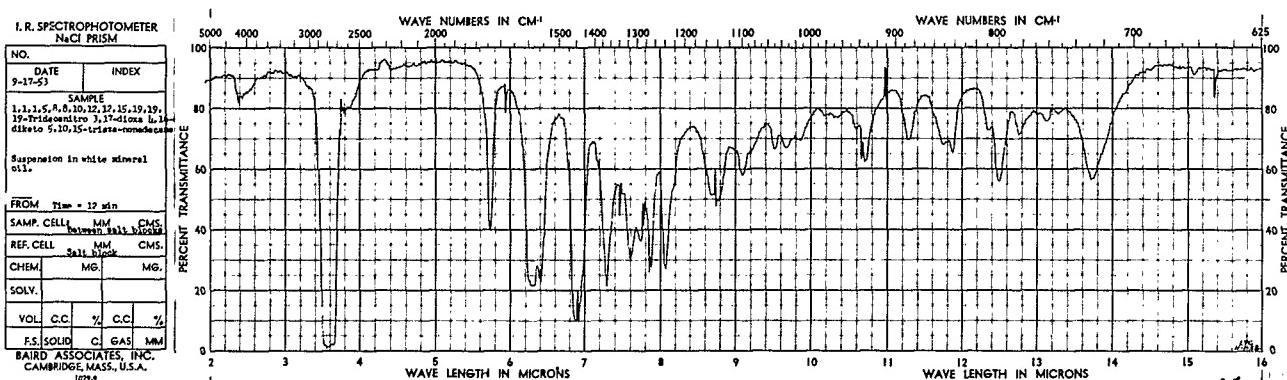
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21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1	g/100 ml H ₂ O at 25°C.	g/100 ml H ₂ O at	°C.
140	g/100 ml	at	25 °C.
<0.1	g/100 ml	(name material used as solvent)	at 25 °C.
		toluene	
		(name material used as solvent)	



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____ : _____

29. Polymerizing properties of the new compound:

- (a) By itself _____
- (b) In mixtures (with additives) _____
- (c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____

30. Availability

- a. Amount now available? _____ research quantities
- b. When was available material first prepared? _____
- c. Amount prepared at that time? _____
- d. Is large production feasible? _____
- e. Plant capacity in existance, lbs/day? _____
- f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 164 Method of Aerojet Report No. 512, p.8.
Calculated ballistic-mortar value = 142

CONFIDENTIAL

Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

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COMPOUND: 2,2,5,9,9,13,16,16-Octanitro-5,13-diaza-

Name 6,12-diketo-7,11-dioxa-heptadecane

Information submitted by:

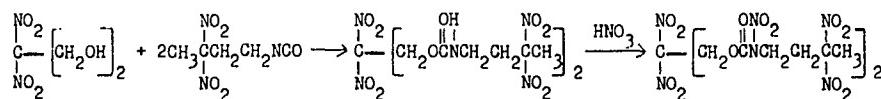
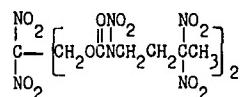
Empirical formula C₁₃H₁₈N₁₀O₂₀

Activity Aerojet-General Corporation

Structure: (configuration)

Person M.B.Frankel and L.T.Carleton

Date 1 November 1953



1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	<u>24.61</u>	<u>2.86</u>		<u>22.08</u>		
By determination	<u>24.78</u>	<u>2.90</u>		<u>21.87</u>		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL OSHXXXXX	<u>2.5-kg weight</u>
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL OSHXXXXXX	<u>48 hr at 100°C</u>
d. Temperature of Explosion	OSRD 3401 p.6	
e. Temperature of Ignition	NOL OSHXXXXXXX	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound _____ New Compound test results
(designation-TNT, Tetryl, N.C., etc.)

a. <u>Tetryl, 32 cm/2.5 kg</u>	<u>137 cm/2.5 kg</u>
b.	<u>1.1 cc/g</u>
c.	
d.	
e.	<u>232°C</u>
f.	
g.	
h.	

4. Heat of formation: $(\Delta H) +$ -2441 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

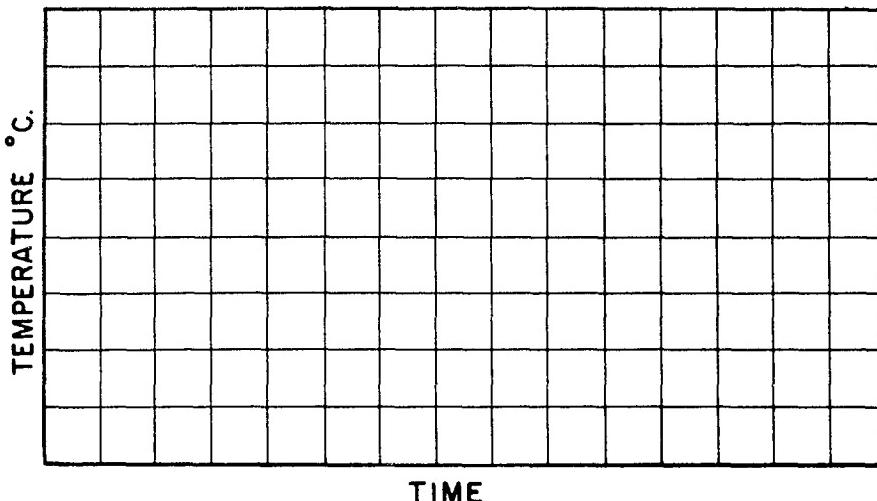
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	By Experiment	By Calculation	Method <small>Description or reference. Separate sheet if necessary.</small>
5. Energy of explosion (Q) <small>(at 25°C. H₂O liquid)</small>	cal/gm		
6. Heat of combustion (H _c) <small>(at 25°C. H₂O liquid)</small>	2511	cal/gm 2527	Aerojet Report No. 417A
7. Specific impulse (I _{sp}) calc:	lb-sec/lb		
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white, crystalline solid		
9. Simple microscope analysis data: <small>(crystal studies)</small>			
10. Density (Macro method) 1.66 gm/cm ³ . <small>(NOL)</small>	(Micro or other method)	gm/cm ³ .	<small>(Explain on separate sheet any unique methods you use.)</small>
11. Index of refraction: (n _D ^{25°C.})	12. Color	white	13. Odor none
14. pH at 25°C. 1.9* <small>(Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.)</small>	With Beckman meter, 0.014 M in acetone/water (5/1 volume ratio)		
15. Hygroscopicity:	New Compound	Reference Compound <small>(designation)</small>	
Visible change on exposure to ambient air.			
% wt Increase by* (a) or (b) <small>(Strike out method (a) or (b) if not used.)</small>	<small>(If other than below methods are used, explain on separate sheet.)</small>		
*Method:			
(a) The sample (approx. 5-10 g. of whole grain small powder or 1 grain of large powder) is placed in a glass weighing bottle. The weighing bottle (cover removed) is placed in a vacuum drying oven for 5 hrs. @ 55°C. Remove weighing bottle from oven, cover with glass stopper, cool in a desiccator and weigh accurately. This is taken as original dry weight of sample. Then place weighing bottle (cover removed) in a humidor (a 10-in. desiccator is a satisfactory vessel) containing 1 liter of 18.6 ± 5% H ₂ SO ₄ . (This gives relative humidity 90 ± 25%). Place in an oven maintained at 30 ± 2°C. On the fourth day remove weighing bottle from the humidor, cover with glass stopper, cool in a desiccator and weigh. Then return to humidor for 24 hours, cool and reweigh. Continue daily weighings until constant weight indicates sample has reached equilibrium with 90% R.H. The % weight increase is then reported as hygroscopicity of the sample.			
(b) An alternate method is in OSRD 3401 p.3.			
16. Volatility: Report as rate of loss in wt % per unit area (for liquid samples measure the surface area; for solids, the sample should be screened between 100-120 mesh U.S. Standard certified sieve) per 4 hrs., after constant rate of loss is obtained during three consecutive 4-hr. periods at (a) 25°C in a vessel through which a stream of dry air is forced, (b) in an oven at 65.5°C or (c) under other test method or conditions.			
<small>(Description or reference to other test or conditions used. Use separate sheet if necessary.)</small>			
Volatility results on:	New Compound	Reference Compound <small>(designation)</small>	
a.	a.		
b.	b.		
c.	c.		
17. Boiling point, or decomposition temperature: <small>(underline which temperature is reported)</small>	°C.		
18. Heat of Vaporization:	g-cal./gm	BTU/lb.	
19. Heat of Fusion:	g-cal./gm	BTU/lb.	
20. Melting point: 117-118 °C.			

*Decreasing slowly.

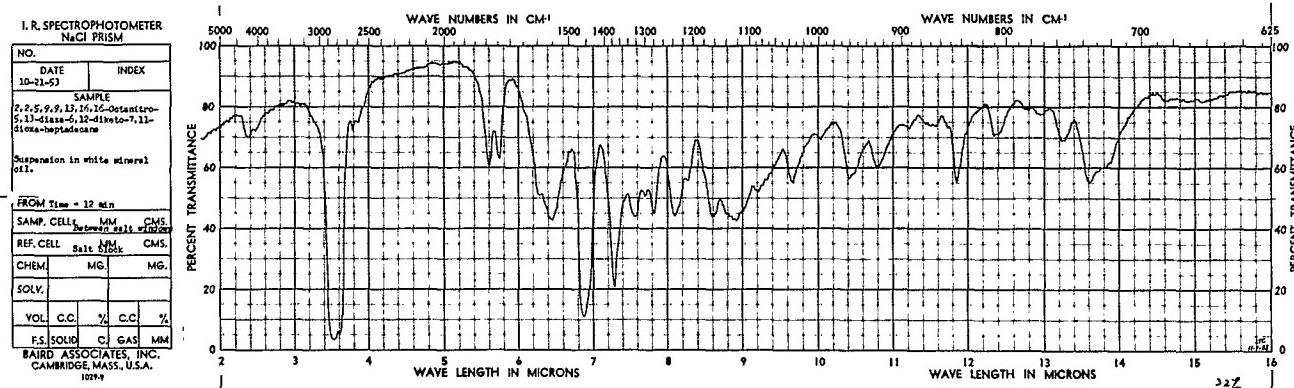
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21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<u><0.1</u>	g/100 ml H ₂ O at 25°C.	g/100 ml H ₂ O at	<u> </u> °C.
<u>73</u>	g/100 ml	acetone	at <u>25</u> °C.
<u>0.1</u>	g/100 ml	toluene	at <u>25</u> °C.
		(name material used as solvent)	
		(name material used as solvent)	



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Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____:

29. Polymerizing properties of the new compound:

- (a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:

Thiokol _____

Methacrylate _____

Other compounds _____

30. Availability

a. Amount now available? research quantities

b. When was available material first prepared?

c. Amount prepared at that time?

d. Is large production feasible?

e. Plant capacity in existence, lbs./day?

f. Outline steps for a quantity production method

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 119

Method of Aerojet Report No. 512, p.8.

Calculated ballistic-mortar value = 128

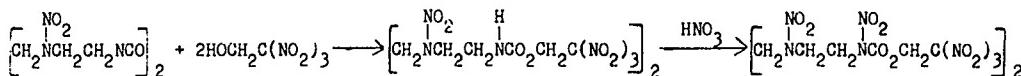
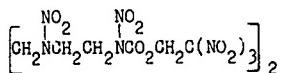
CONFIDENTIAL

Date Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,1,1,5,8,11,14,18,18,18-Decanitro-3,16-

Name dioxa-1,15-diketo-5,8,11,14-tetraza- Information submitted by:
 Empirical formula octadecane C₁₂H₁₆N₄O₂₄ Activity Aerojet-General Corporation
 Structure: (configuration) Person M.B.Frankel and L.T.Carleton
 Date 1 November 1953



1. Quantitative analysis: (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	<u>19.47</u>	<u>2.18</u>		<u>26.49</u>		
By determination	<u>19.51</u>	<u>2.29</u>		<u>25.78</u>		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL XOSRD XXXXK	<u>2.5-kg weight</u>
b. Thermal Stability	OSRD 3401 p.8	<u>48 hr at 100°C</u>
c. Vacuum Stability	XOSRD XXXXK	
d. Temperature of Explosion	OSRD 3401 p.6	
e. Temperature of Ignition	OSRD XXXXK	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	<u>20 cm/2.5 kg</u>
b.	<u>22.5 cc/g</u>
c.	
d.	
e.	<u>213°C</u>
f.	
g.	
h.	

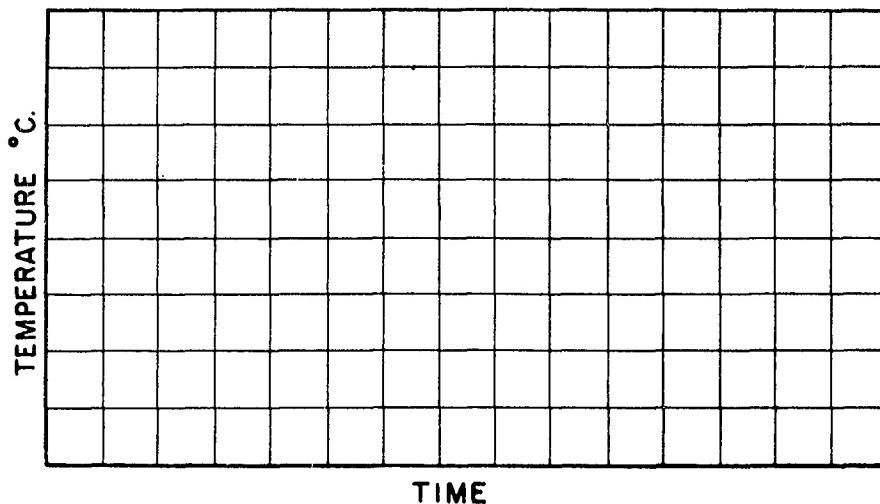
4. Heat of formation: (ΔH) -190 (Indicate sign) Kg. calories at 25°C., 1 atm. pressure

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	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)	_____	cal/gm _____	_____
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2006	cal/gm 2086	Aerojet Report No. 117A
7. Specific impulse (I _{sp}) calc:	_____	lb-sec/lb _____	_____
8. Physical form of compound (viscous liquid, crystalline type, etc.)	white, crystalline solid		
9. Simple microscope analysis data: (crystal studies)	_____		
10. Density (Macro method) 1.78 gm/cm ³ . (NOL)	(Micro or other method) _____ gm/cm ³ . (Explain on separate sheet any unique methods you use.)		
11. Index of refraction: (n _D ^{25°C.})	_____	12. Color white	13. Odor none
14. pH at 25°C. 2.1	(Method reference OSRD 3401 p.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) With Beckman meter, 0.0058 M in acetone/water (5/1 volume ratio).		
15. Hygroscopicity:	New Compound	Reference Compound (designation)	
Visible change on exposure to ambient air.	_____		
% wt Increase by* (a) or (b) (Strike out method (a) or (b) if not used.)	(If other than below methods are used, explain on separate sheet.)		
*Method:			
(a) The sample (approx. 5-10 g. of whole grain small powder or 1 grain of large powder) is placed in a glass weighing bottle. The weighing bottle (cover removed) is placed in a vacuum drying oven for 5 hrs. @ 55°C. Remove weighing bottle from oven, cover with glass stopper, cool in a desiccator and weigh accurately. This is taken as original dry weight of sample. Then place weighing bottle (cover removed) in a humidior (a 10-in. desiccator is a satisfactory vessel) containing 1 liter of 18.6 ± .5% H ₂ SO ₄ . (This gives relative humidity 90 ± .25%). Place in an oven maintained at 30 ± 2°C. On the fourth day remove weighing bottle from the humidior, cover with glass stopper, cool in a desiccator and weigh. Then return to humidior for 24 hours, cool and reweigh. Continue daily weighings until constant weight indicates sample has reached equilibrium with 90% R.H. The % weight increase is then reported as hygroscopicity of the sample.			
(b) An alternate method is in OSRD 3401 p.3.			
16. Volatility: Report as rate of loss in wt % per unit area (for liquid samples measure the surface area; for solids, the sample should be screened between 100-120 mesh U.S. Standard certified sieve) per 4 hrs., after constant rate of loss is obtained during three consecutive 4-hr. periods at (a) 25°C in a vessel through which a stream of dry air is forced, (b) in an oven at 65.5°C or (c) under other test method or conditions.			
(Description or reference to other test or conditions used. Use separate sheet if necessary.)			
Volatility results on:	New Compound	Reference Compound (designation)	
a.	_____	a.	_____
b.	_____	b.	_____
c.	_____	c.	_____
17. Boiling point, or decomposition temperature: (underline which temperature is reported)	_____ °C.		
18. Heat of Vaporization:	_____ g-cal./gm	BTU/lb.	
19. Heat of Fusion:	_____ g-cal./gm	BTU/lb.	
20. Melting point:	127-128 °C.		

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21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1 g/100 ml H₂O at 25°C. g/100 ml H₂O at _____ °C.
28 g/100 ml acetone at 25 °C.
0.1 g/100 ml toluene at 25 °C.
(name material used as solvent)

I.R. SPECTROPHOTOMETER
NaCl PRISM

NO.	DATE	INDEX
10-21-53		

SAMPLE
1,1,11,15,15-penta-1,3,15,15-Dinitro-3,15-dimethyl-1,15-diket-5,8,11,13-tetracos-octadecane
Suspension in white mineral oil.

FROM Time = 12 min

SAMP. CELL Between salt CMS

REF. CELL Salt block CMS

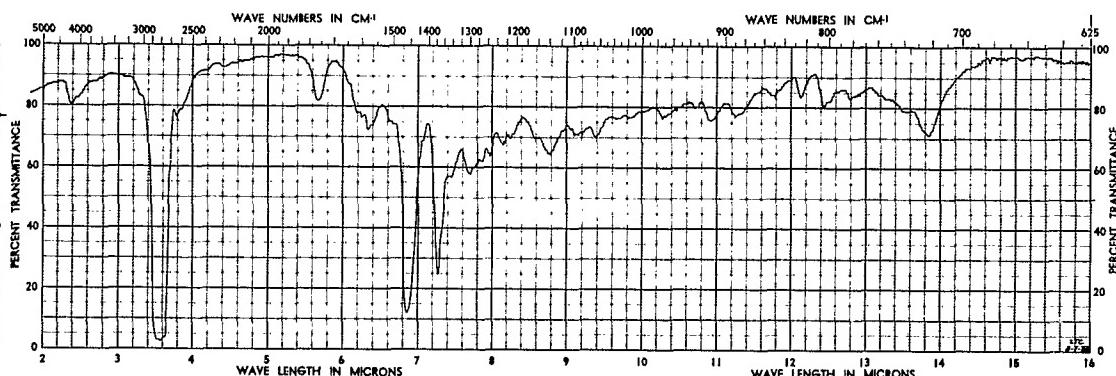
CHRM. MG. MG.

SOLV.

VOL. C.C. % C.C. %

F.S. SOLID C. GAS MM

BAIRD ASSOCIATES, INC.
CAMBRIDGE, MASS., U.S.A.
1959



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Report No. 770
SPIA/M3

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____: _____

29. Polymerizing properties of the new compound:

- (a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:

Thiokol _____

Methacrylate _____

Other compounds _____

30. Availability

- a. Amount now available? research quantities
b. When was available material first prepared?
c. Amount prepared at that time?
d. Is large production feasible?
e. Plant capacity in existence, lbs./day?
f. Outline steps for a quantity production method
- _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 11.2

Calculated ballistic-mortar value = 14.1

{ Method of Aerojet Report No. 512, p.8.

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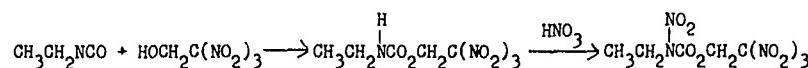
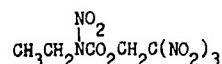
Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,1,1,5-Tetranitro 3-oxa 4-keto

Name 5-aza-heptane
Empirical formula C₅H₂N₅O₁₀
Structure: (configuration)

Information submitted by:
Activity Aerojet-General Corporation
Person M.B.Frankel and L.T.Carleton
Date 1 November 1953



1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	20.21	2.38		23.57		
By determination	20.55	2.04		23.68		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

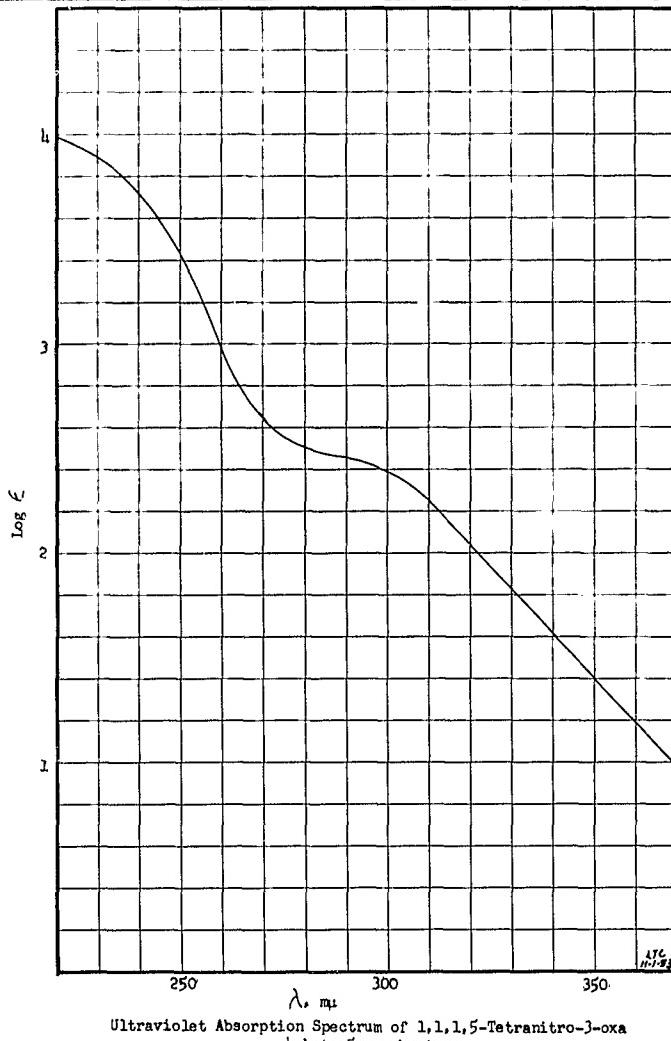
Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD XXXX	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL OSRD XXXX	48 hr at 100°C
d. Temperature of Explosion	OSRD 3401 p.6	
e. Temperature of Ignition	NOL OSRD XXXX	
f.		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	19 cm/2.5 kg
b.	28.4 cc/g
c.	
d.	
e.	235°C
f.	
g.	
h.	

4. Heat of formation: (ΔH) + -106 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2031	cal/gm	2032
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	crystalline, white plates		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method) (NOL)	1.69 gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
14. pH at 25°C. 3.9*	(Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) With Beckman meter, 0.03 M in acetone/water (5/1 volume ratio)		

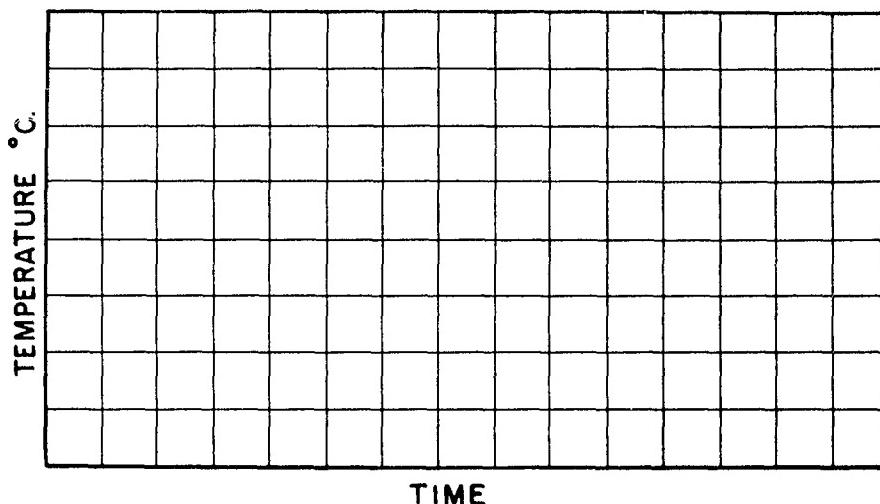


*Decreasing
Page 58
Appendix

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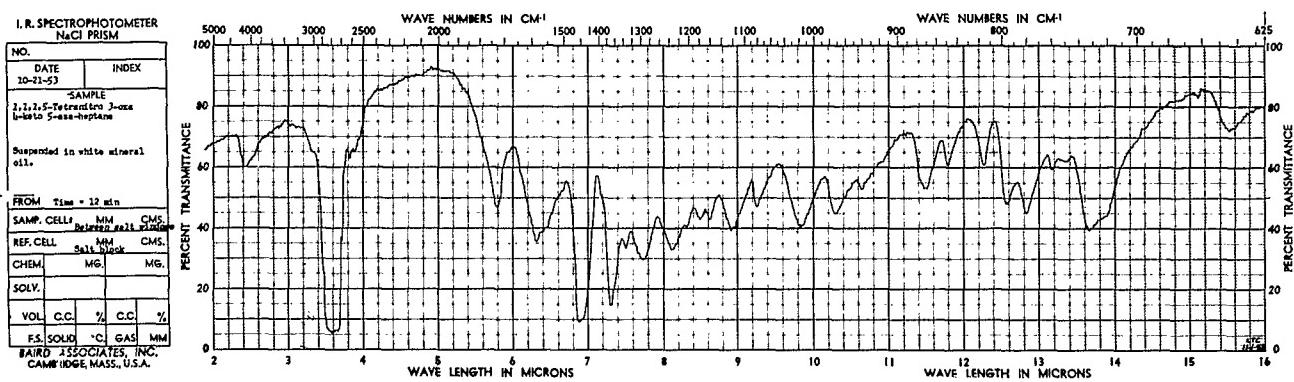
SPIA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<u><0.1</u>	g/100 ml H ₂ O at 25°C.	<u> </u> g/100 ml H ₂ O at <u> </u> °C.
<u>310</u>	g/100 ml <u>acetone</u> (name material used as solvent)	at <u>25</u> °C.
<u>65</u>	g/100 ml <u>toluene</u> (name material used as solvent)	at <u>25</u> °C.



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? _____ research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____
Calculated lead-block value = 116) Method of Aerojet Report No. 512, p.8
Calculated ballistic-mortar value = 136)

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Date Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

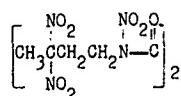
Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: N,N'-Dinitro-N,N'-bis(3,3-Dinitrobutyl)

Name Oxamide

Empirical formula C₁₀H₁₄N₈O₇

Structure: (configuration)

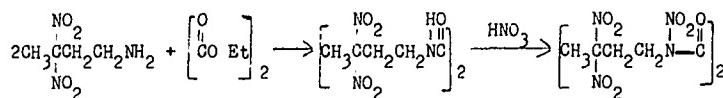


Information submitted by:

Activity Aerojet-General Corporation

Person M.B.Frankel and L.T.Carleton

Date 1 November 1953



1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	25.54	3.00		23.83		
By determination	25.84	3.26		24.37		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method
a. Impact Sensitivity	OSRD 3185
b. Thermal Stability	OSRD 3401 p.8
c. Vacuum Stability	OSRD 3401 p.10
d. Temperature of Explosion	OSRD 3401 p.6
e. Temperature of Ignition	OSRD 3401 p.6
f.	
g.	
h.	

RESULTS OF ABOVE TESTS

Reference compound _____
(designation-TNT, Tetryl, N.C., etc.)

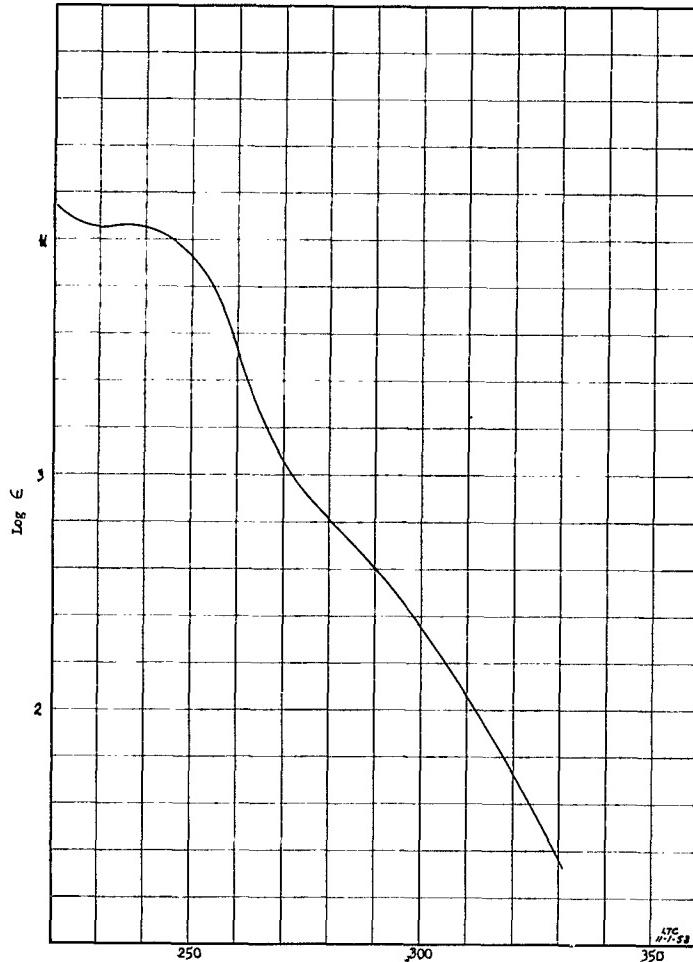
New Compound test results

a.	_____
b.	_____
c.	_____
d.	_____
e.	_____
f.	_____
g.	_____
h.	_____

**4. Heat of formation: (ΔH) -115 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)**

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	By Experiment	By Calculation	Method
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	Description or reference. Separate sheet if necessary.
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2708	cal/gm	2773
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	flat white crystals		
9. Simple microscope analysis data: (crystal studies)			
10. Density(Macro method)	gm/cm ³ .	(Micro or other method)	gm/cm ³ . (Explain on separate sheet any unique methods you use.)
11. Index of refraction: (n _D ^{25°C.})		12. Color	white
14. pH at 25°C. 14.8*	(Method reference OSRD 3401 c.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator paper or Beckman pH meter.) With Beckman ph meter, 0.001 M in acetone/water (5/1 volume ratio)		



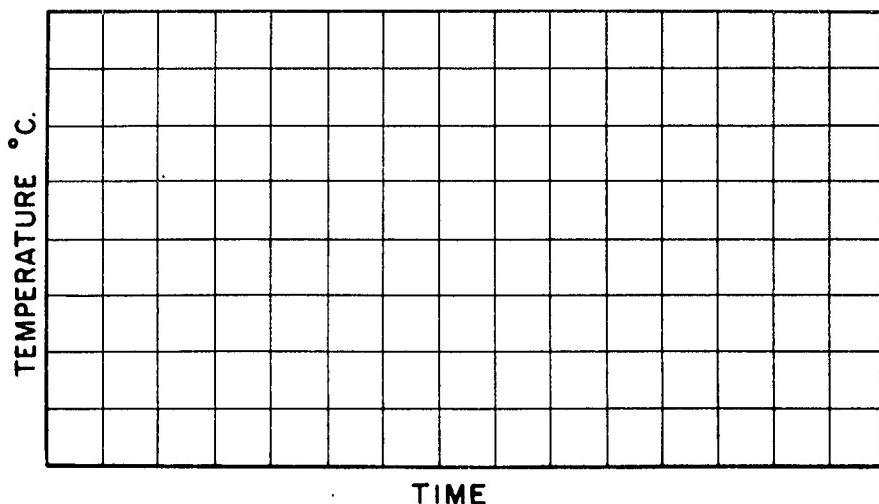
Ultraviolet Absorption Spectrum of N,N'-Dinitro-bis N,N'-
(3,3-Dinitrobutyl) Oxamide in Methanol

20. Melting point: 142-143 °C.

*Decreasing steadily.

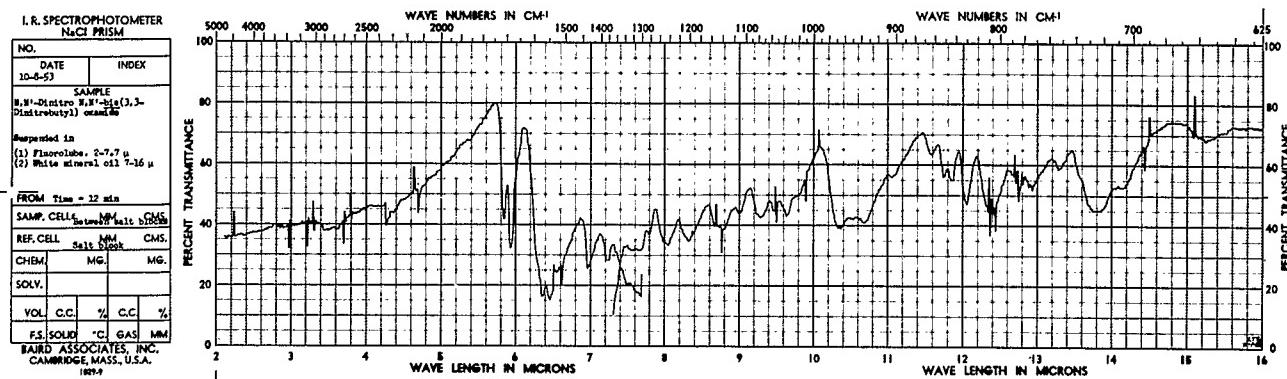
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21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<u><0.1</u>	g/100 ml H ₂ O at 25°C.	<u> </u> g/100 ml H ₂ O at <u> </u> °C.
<u>46</u>	g/100 ml <u>acetone</u> (name material used as solvent)	at <u>25</u> °C.
<u>0.3</u>	g/100 ml <u>toluene</u> (name material used as solvent)	at <u>25</u> °C.



CONFIDENTIALReport No. 770
SPLA/M3

Under compatability we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatability with ethyl cellulose: _____

26. Compatability with nitrocellulose: _____

27. Compatability with rubber: _____

28. Compatability with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____

30. Availability
a. Amount now available? research quantities _____
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated lead-block value = 118 ; Method of Aerojet Report No. 512, p.8
Calculated ballistic-mortar value = 129)

